

=> FIL WPIX  
 FILE 'WPIX' ENTERED AT 09:21:35 ON 17 DEC 2009  
 COPYRIGHT (C) 2009 THOMSON REUTERS

'BI ABEX' IS DEFAULT SEARCH FIELD FOR 'WPIX' FILE

=> D HIS NOFILE

FILE 'WPIX' ENTERED AT 08:34:33 ON 17 DEC 2009  
 E AZNARP/AU  
 E AZNAR P/AU

L1	4	SEA	SPE=ON	ABB=ON	PLU=ON	"AZNAR P"/AU
L2	1152887	SEA	SPE=ON	ABB=ON	PLU=ON	COLUMN?/BI, ABEX OR TUBE/BI, ABEX OR TUBES/BI, ABEX OR TUBULAR?/BI, ABEX
L3	98074	SEA	SPE=ON	ABB=ON	PLU=ON	CHROMATOG?/BI, ABEX OR LPLC/BI, A BEX OR MPLC/BI, ABEX
L4	348485	SEA	SPE=ON	ABB=ON	PLU=ON	SILICA?/BI, ABEX OR SILICON?/BI, ABEX (2A) DIOXIDE?/BI, ABEX OR SIO2/BI, ABEX
L5	796207	SEA	SPE=ON	ABB=ON	PLU=ON	?SPHER?/BI, ABEX OR ?BEAD?/BI, AB EX OR ?BALL?/BI, ABEX
L6	233309	SEA	SPE=ON	ABB=ON	PLU=ON	?POROS?/BI, ABEX OR ?POROUS?/BI, ABEX
L7	100323	SEA	SPE=ON	ABB=ON	PLU=ON	FLASH?/BI, ABEX
L8	218468	SEA	SPE=ON	ABB=ON	PLU=ON	GEL###/BI, ABEX
L9	301190	SEA	SPE=ON	ABB=ON	PLU=ON	MICRON?/BI, ABEX OR ANGSTROM?/BI , ABEX
L10	37404	SEA	SPE=ON	ABB=ON	PLU=ON	L2 AND L3
L11	12170	SEA	SPE=ON	ABB=ON	PLU=ON	L10 AND L4
L12	920	SEA	SPE=ON	ABB=ON	PLU=ON	L11 AND L6
L13	210	SEA	SPE=ON	ABB=ON	PLU=ON	L12 AND L5
L14	115	SEA	SPE=ON	ABB=ON	PLU=ON	L13 AND L8
L15	39	SEA	SPE=ON	ABB=ON	PLU=ON	L14 AND L9
L16	2	SEA	SPE=ON	ABB=ON	PLU=ON	L15 AND L7
L17	2	SEA	SPE=ON	ABB=ON	PLU=ON	L10 AND L1
L18	39	SEA	SPE=ON	ABB=ON	PLU=ON	L15 OR L16
L19	38	SEA	SPE=ON	ABB=ON	PLU=ON	L18 NOT L17
L20	35265	SEA	SPE=ON	ABB=ON	PLU=ON	B01J0020/IPC
L21	23	SEA	SPE=ON	ABB=ON	PLU=ON	L19 AND L20
L22	3635	SEA	SPE=ON	ABB=ON	PLU=ON	C01B0033-12/IPC
L23	3	SEA	SPE=ON	ABB=ON	PLU=ON	L19 AND L22
L24	23	SEA	SPE=ON	ABB=ON	PLU=ON	L21 OR L23
L25	15	SEA	SPE=ON	ABB=ON	PLU=ON	L19 NOT L24
L26	16	SEA	SPE=ON	ABB=ON	PLU=ON	1808-2002/PY, PRY, AY AND L24
L27	9	SEA	SPE=ON	ABB=ON	PLU=ON	1808-2002/PRY, PY, AY AND L25
L28	68875	SEA	SPE=ON	ABB=ON	PLU=ON	(B05-B02C OR B11-B OR B11-C OR C05-B02C OR C11-B OR C11-C OR J01-D01A OR J04-B01C OR S03-E09C5)/MC
L29	13616	SEA	SPE=ON	ABB=ON	PLU=ON	L28 AND L2
L30	9032	SEA	SPE=ON	ABB=ON	PLU=ON	L29 AND L3
L31	1504	SEA	SPE=ON	ABB=ON	PLU=ON	L30 AND L4
L32	380	SEA	SPE=ON	ABB=ON	PLU=ON	L31 AND L6
L33	119	SEA	SPE=ON	ABB=ON	PLU=ON	L32 AND L5
L34	55	SEA	SPE=ON	ABB=ON	PLU=ON	L33 AND L8
L35	27	SEA	SPE=ON	ABB=ON	PLU=ON	L34 AND L9
L36	1	SEA	SPE=ON	ABB=ON	PLU=ON	L35 AND L1
L37	2	SEA	SPE=ON	ABB=ON	PLU=ON	L36 OR L17
L38	26	SEA	SPE=ON	ABB=ON	PLU=ON	L35 NOT L37
L39	18	SEA	SPE=ON	ABB=ON	PLU=ON	1808-2002/PY, PRY, AY AND L38

L40 21 SEA SPE=ON ABB=ON PLU=ON L39 OR L26  
 L41 4 SEA SPE=ON ABB=ON PLU=ON L27 NOT (L40 OR L37)  
  
 FILE 'COMPENDEX', DISSABS, INSPEC, NTIS, PASCAL' ENTERED AT 09:04:34  
 ON 17 DEC 2009  
 FILE 'COMPENDEX'  
 L42 520835 SEA SPE=ON ABB=ON PLU=ON COLUMN?/BI,ABEX OR TUBE/BI,ABEX  
 OR TUBES/BI,ABEX OR TUBULAR?/BI,ABEX  
 FILE 'DISSABS'  
 L43 30377 SEA SPE=ON ABB=ON PLU=ON COLUMN?/BI,ABEX OR TUBE/BI,ABEX  
 OR TUBES/BI,ABEX OR TUBULAR?/BI,ABEX  
 FILE 'INSPEC'  
 L44 245619 SEA SPE=ON ABB=ON PLU=ON COLUMN?/BI,ABEX OR TUBE/BI,ABEX  
 OR TUBES/BI,ABEX OR TUBULAR?/BI,ABEX  
 FILE 'NTIS'  
 L45 61371 SEA SPE=ON ABB=ON PLU=ON COLUMN?/BI,ABEX OR TUBE/BI,ABEX  
 OR TUBES/BI,ABEX OR TUBULAR?/BI,ABEX  
 FILE 'PASCAL'  
 L46 297843 SEA SPE=ON ABB=ON PLU=ON COLUMN?/BI,ABEX OR TUBE/BI,ABEX  
 OR TUBES/BI,ABEX OR TUBULAR?/BI,ABEX  
 TOTAL FOR ALL FILES  
 L47 1156045 SEA SPE=ON ABB=ON PLU=ON L2  
 FILE 'COMPENDEX'  
 L48 104890 SEA SPE=ON ABB=ON PLU=ON CHROMATOG?/BI,ABEX OR LPLC/BI,ABEX  
 OR MPLC/BI,ABEX  
 FILE 'DISSABS'  
 L49 21294 SEA SPE=ON ABB=ON PLU=ON CHROMATOG?/BI,ABEX OR LPLC/BI,ABEX  
 OR MPLC/BI,ABEX  
 FILE 'INSPEC'  
 L50 15016 SEA SPE=ON ABB=ON PLU=ON CHROMATOG?/BI,ABEX OR LPLC/BI,ABEX  
 OR MPLC/BI,ABEX  
 FILE 'NTIS'  
 L51 16055 SEA SPE=ON ABB=ON PLU=ON CHROMATOG?/BI,ABEX OR LPLC/BI,ABEX  
 OR MPLC/BI,ABEX  
 FILE 'PASCAL'  
 L52 290739 SEA SPE=ON ABB=ON PLU=ON CHROMATOG?/BI,ABEX OR LPLC/BI,ABEX  
 OR MPLC/BI,ABEX  
 TOTAL FOR ALL FILES  
 L53 447994 SEA SPE=ON ABB=ON PLU=ON L3  
 FILE 'COMPENDEX'  
 L54 171151 SEA SPE=ON ABB=ON PLU=ON SILICA?/BI,ABEX OR SILICON?/BI,ABEX  
 (2A) DIOXIDE?/BI,ABEX OR SIO2/BI,ABEX  
 FILE 'DISSABS'  
 L55 11639 SEA SPE=ON ABB=ON PLU=ON SILICA?/BI,ABEX OR SILICON?/BI,ABEX  
 (2A) DIOXIDE?/BI,ABEX OR SIO2/BI,ABEX  
 FILE 'INSPEC'  
 L56 163790 SEA SPE=ON ABB=ON PLU=ON SILICA?/BI,ABEX OR SILICON?/BI,ABEX  
 (2A) DIOXIDE?/BI,ABEX OR SIO2/BI,ABEX  
 FILE 'NTIS'  
 L57 15796 SEA SPE=ON ABB=ON PLU=ON SILICA?/BI,ABEX OR SILICON?/BI,ABEX  
 (2A) DIOXIDE?/BI,ABEX OR SIO2/BI,ABEX  
 FILE 'PASCAL'  
 L58 141968 SEA SPE=ON ABB=ON PLU=ON SILICA?/BI,ABEX OR SILICON?/BI,ABEX  
 (2A) DIOXIDE?/BI,ABEX OR SIO2/BI,ABEX  
 TOTAL FOR ALL FILES  
 L59 504344 SEA SPE=ON ABB=ON PLU=ON L4  
 FILE 'COMPENDEX'  
 L60 560023 SEA SPE=ON ABB=ON PLU=ON ?SPHER?/BI,ABEX OR ?BEAD?/BI,ABEX  
 OR ?BALL?/BI,ABEX  
 FILE 'DISSABS'

L61	76885	SEA SPE=ON	ABB=ON	PLU=ON	?SPHER?/BI, ABEX OR ?BEAD?/BI, ABEX OR ?BALL?/BI, ABEX
	FILE 'INSPEC'				
L62	717279	SEA SPE=ON	ABB=ON	PLU=ON	?SPHER?/BI, ABEX OR ?BEAD?/BI, ABEX OR ?BALL?/BI, ABEX
	FILE 'NTIS'				
L63	158415	SEA SPE=ON	ABB=ON	PLU=ON	?SPHER?/BI, ABEX OR ?BEAD?/BI, ABEX OR ?BALL?/BI, ABEX
	FILE 'PASCAL'				
L64	337558	SEA SPE=ON	ABB=ON	PLU=ON	?SPHER?/BI, ABEX OR ?BEAD?/BI, ABEX OR ?BALL?/BI, ABEX
	TOTAL FOR ALL FILES				
L65	1850160	SEA SPE=ON	ABB=ON	PLU=ON	L5
	FILE 'COMPEINDEX'				
L66	167714	SEA SPE=ON	ABB=ON	PLU=ON	?POROS?/BI, ABEX OR ?POROUS?/BI, ABEX
	FILE 'DISSABS'				
L67	14020	SEA SPE=ON	ABB=ON	PLU=ON	?POROS?/BI, ABEX OR ?POROUS?/BI, ABEX
	FILE 'INSPEC'				
L68	101429	SEA SPE=ON	ABB=ON	PLU=ON	?POROS?/BI, ABEX OR ?POROUS?/BI, ABEX
	FILE 'NTIS'				
L69	18617	SEA SPE=ON	ABB=ON	PLU=ON	?POROS?/BI, ABEX OR ?POROUS?/BI, ABEX
	FILE 'PASCAL'				
L70	160390	SEA SPE=ON	ABB=ON	PLU=ON	?POROS?/BI, ABEX OR ?POROUS?/BI, ABEX
	TOTAL FOR ALL FILES				
L71	462170	SEA SPE=ON	ABB=ON	PLU=ON	L6
	FILE 'COMPEINDEX'				
L72	30860	SEA SPE=ON	ABB=ON	PLU=ON	FLASH?/BI, ABEX
	FILE 'DISSABS'				
L73	3749	SEA SPE=ON	ABB=ON	PLU=ON	FLASH?/BI, ABEX
	FILE 'INSPEC'				
L74	39129	SEA SPE=ON	ABB=ON	PLU=ON	FLASH?/BI, ABEX
	FILE 'NTIS'				
L75	8020	SEA SPE=ON	ABB=ON	PLU=ON	FLASH?/BI, ABEX
	FILE 'PASCAL'				
L76	22251	SEA SPE=ON	ABB=ON	PLU=ON	FLASH?/BI, ABEX
	TOTAL FOR ALL FILES				
L77	104009	SEA SPE=ON	ABB=ON	PLU=ON	L7
	FILE 'COMPEINDEX'				
L78	105103	SEA SPE=ON	ABB=ON	PLU=ON	GEL####/BI, ABEX
	FILE 'DISSABS'				
L79	20390	SEA SPE=ON	ABB=ON	PLU=ON	GEL####/BI, ABEX
	FILE 'INSPEC'				
L80	60192	SEA SPE=ON	ABB=ON	PLU=ON	GEL####/BI, ABEX
	FILE 'NTIS'				
L81	8255	SEA SPE=ON	ABB=ON	PLU=ON	GEL####/BI, ABEX
	FILE 'PASCAL'				
L82	204422	SEA SPE=ON	ABB=ON	PLU=ON	GEL####/BI, ABEX
	TOTAL FOR ALL FILES				
L83	398362	SEA SPE=ON	ABB=ON	PLU=ON	L8
	FILE 'COMPEINDEX'				
L84	52733	SEA SPE=ON	ABB=ON	PLU=ON	MICRON?/BI, ABEX OR ANGSTROM?/BI, ABEX
	FILE 'DISSABS'				
L85	7862	SEA SPE=ON	ABB=ON	PLU=ON	MICRON?/BI, ABEX OR ANGSTROM?/BI, ABEX

```

FILE 'INSPEC'
L86 155696 SEA SPE=ON ABB=ON PLU=ON MICRON?/BI, ABEX OR ANGSTROM?/BI
, ABEX
FILE 'NTIS'
L87 13577 SEA SPE=ON ABB=ON PLU=ON MICRON?/BI, ABEX OR ANGSTROM?/BI
, ABEX
FILE 'PASCAL'
L88 39957 SEA SPE=ON ABB=ON PLU=ON MICRON?/BI, ABEX OR ANGSTROM?/BI
, ABEX
TOTAL FOR ALL FILES
L89 269825 SEA SPE=ON ABB=ON PLU=ON L9

FILE 'STNGUIDE' ENTERED AT 09:07:38 ON 17 DEC 2009

FILE 'COMPENDEX, DISSABS, INSPEC, NTIS, PASCAL' ENTERED AT 09:15:39
ON 17 DEC 2009
FILE 'COMPENDEX'
L90 23338 SEA SPE=ON ABB=ON PLU=ON L42 AND L48
FILE 'DISSABS'
L91 4696 SEA SPE=ON ABB=ON PLU=ON L43 AND L49
FILE 'INSPEC'
L92 2104 SEA SPE=ON ABB=ON PLU=ON L44 AND L50
FILE 'NTIS'
L93 3454 SEA SPE=ON ABB=ON PLU=ON L45 AND L51
FILE 'PASCAL'
L94 49396 SEA SPE=ON ABB=ON PLU=ON L46 AND L52
TOTAL FOR ALL FILES
L95 82988 SEA SPE=ON ABB=ON PLU=ON L47 AND L53
FILE 'COMPENDEX'
L96 3239 SEA SPE=ON ABB=ON PLU=ON L90 AND L54
FILE 'DISSABS'
L97 573 SEA SPE=ON ABB=ON PLU=ON L91 AND L55
FILE 'INSPEC'
L98 186 SEA SPE=ON ABB=ON PLU=ON L92 AND L56
FILE 'NTIS'
L99 322 SEA SPE=ON ABB=ON PLU=ON L93 AND L57
FILE 'PASCAL'
L100 5736 SEA SPE=ON ABB=ON PLU=ON L94 AND L58
TOTAL FOR ALL FILES
L101 10056 SEA SPE=ON ABB=ON PLU=ON L95 AND L59
FILE 'COMPENDEX'
L102 382 SEA SPE=ON ABB=ON PLU=ON L96 AND L66
FILE 'DISSABS'
L103 86 SEA SPE=ON ABB=ON PLU=ON L97 AND L67
FILE 'INSPEC'
L104 25 SEA SPE=ON ABB=ON PLU=ON L98 AND L68
FILE 'NTIS'
L105 16 SEA SPE=ON ABB=ON PLU=ON L99 AND L69
FILE 'PASCAL'
L106 598 SEA SPE=ON ABB=ON PLU=ON L100 AND L70
TOTAL FOR ALL FILES
L107 1107 SEA SPE=ON ABB=ON PLU=ON L101 AND L71
FILE 'COMPENDEX'
L108 91 SEA SPE=ON ABB=ON PLU=ON L102 AND L60
FILE 'DISSABS'
L109 22 SEA SPE=ON ABB=ON PLU=ON L103 AND L61
FILE 'INSPEC'
L110 2 SEA SPE=ON ABB=ON PLU=ON L104 AND L62
FILE 'NTIS'
L111 3 SEA SPE=ON ABB=ON PLU=ON L105 AND L63

```

FILE 'PASCAL'  
 L112 124 SEA SPE=ON ABB=ON PLU=ON L106 AND L64  
 TOTAL FOR ALL FILES  
 L113 242 SEA SPE=ON ABB=ON PLU=ON L107 AND L65  
 FILE 'COMPENDEX'  
 L114 32 SEA SPE=ON ABB=ON PLU=ON L108 AND L78  
 FILE 'DISSABS'  
 L115 7 SEA SPE=ON ABB=ON PLU=ON L109 AND L79  
 FILE 'INSPEC'  
 L116 0 SEA SPE=ON ABB=ON PLU=ON L110 AND L80  
 FILE 'NTIS'  
 L117 1 SEA SPE=ON ABB=ON PLU=ON L111 AND L81  
 FILE 'PASCAL'  
 L118 45 SEA SPE=ON ABB=ON PLU=ON L112 AND L82  
 TOTAL FOR ALL FILES  
 L119 85 SEA SPE=ON ABB=ON PLU=ON L113 AND L83  
 FILE 'COMPENDEX'  
 L120 1 SEA SPE=ON ABB=ON PLU=ON L114 AND L84  
 FILE 'DISSABS'  
 L121 0 SEA SPE=ON ABB=ON PLU=ON L115 AND L85  
 FILE 'INSPEC'  
 L122 0 SEA SPE=ON ABB=ON PLU=ON L116 AND L86  
 FILE 'NTIS'  
 L123 0 SEA SPE=ON ABB=ON PLU=ON L117 AND L87  
 FILE 'PASCAL'  
 L124 3 SEA SPE=ON ABB=ON PLU=ON L118 AND L88  
 TOTAL FOR ALL FILES  
 L125 4 SEA SPE=ON ABB=ON PLU=ON L119 AND L89  
 FILE 'COMPENDEX'  
 L126 1 SEA SPE=ON ABB=ON PLU=ON L108 AND L84  
 FILE 'DISSABS'  
 L127 4 SEA SPE=ON ABB=ON PLU=ON L109 AND L85  
 FILE 'INSPEC'  
 L128 0 SEA SPE=ON ABB=ON PLU=ON L110 AND L86  
 FILE 'NTIS'  
 L129 0 SEA SPE=ON ABB=ON PLU=ON L111 AND L87  
 FILE 'PASCAL'  
 L130 5 SEA SPE=ON ABB=ON PLU=ON L112 AND L88  
 TOTAL FOR ALL FILES  
 L131 10 SEA SPE=ON ABB=ON PLU=ON L113 AND L89  
 FILE 'COMPENDEX'  
 L132 1 SEA SPE=ON ABB=ON PLU=ON L120 OR L126  
 FILE 'DISSABS'  
 L133 4 SEA SPE=ON ABB=ON PLU=ON L121 OR L127  
 FILE 'INSPEC'  
 L134 0 SEA SPE=ON ABB=ON PLU=ON L122 OR L128  
 FILE 'NTIS'  
 L135 0 SEA SPE=ON ABB=ON PLU=ON L123 OR L129  
 FILE 'PASCAL'  
 L136 5 SEA SPE=ON ABB=ON PLU=ON L124 OR L130  
 TOTAL FOR ALL FILES  
 L137 10 SEA SPE=ON ABB=ON PLU=ON L125 OR L131  
 L138 9 DUP REM L137 (1 DUPLICATE REMOVED)  
 ANSWER '1' FROM FILE COMPENDEX  
 ANSWERS '2-5' FROM FILE DISSABS  
 ANSWERS '6-9' FROM FILE PASCAL

FILE 'COMPENDEX'

L139 1 SEA L138  
 L140 0 SEA SPE=ON ABB=ON PLU=ON 1808-2002/PY,PRY,AY AND L139

```

FILE 'DISSABS'
L141      4 SEA L138
L142      2 SEA SPE=ON  ABB=ON  PLU=ON  1808-2002/PY,PRY,AY AND L141
FILE 'INSPEC'
L143      0 SEA L138
L144      0 SEA SPE=ON  ABB=ON  PLU=ON  1808-2002/PY,PRY,AY AND L143
FILE 'NTIS'
L145      0 SEA L138
L146      0 SEA SPE=ON  ABB=ON  PLU=ON  1808-2002/PY,PRY,AY AND L145
FILE 'PASCAL'
L147      4 SEA L138
L148      3 SEA SPE=ON  ABB=ON  PLU=ON  1808-2002/PY,PRY,AY AND L147
TOTAL FOR ALL FILES
L149      5 SEA SPE=ON  ABB=ON  PLU=ON  1808-2002/PY,PRY,AY AND L138

FILE 'DISSABS, PASCAL' ENTERED AT 09:19:49 ON 17 DEC 2009
FILE 'DISSABS'
L150      2 SEA SPE=ON  ABB=ON  PLU=ON  1808-2002/PY,PRY,AY AND L141
FILE 'PASCAL'
L151      3 SEA SPE=ON  ABB=ON  PLU=ON  1808-2002/PY,PRY,AY AND L147
TOTAL FOR ALL FILES
L152      5 SEA SPE=ON  ABB=ON  PLU=ON  L149

```

FILE 'WPIX' ENTERED AT 09:21:35 ON 17 DEC 2009

=> D L37 1-2 IFULL

```

L37  ANSWER 1 OF 2  WPIX COPYRIGHT 2009      THOMSON REUTERS on STN
ACCESSION NUMBER:    2003-579832 [55]  WPIX
DOC. NO. CPI:        C2003-157116 [55]
DOC. NO. NON-CPI:    N2003-460971 [55]
TITLE:
Flash chromatography or solid phase
extraction columns having good performance
in separation of e.g. pharmaceutical compounds,
filled with porous, spherical or
semi-spherical silica gel
DERWENT CLASS:       B05; C03; J01; J04; S03
INVENTOR:            AZNAR P
PATENT ASSIGNEE:     (AZNA-I) AZNAR P
COUNTRY COUNT:       100

```

#### PATENT INFORMATION:

PATENT NO	KIND	DATE	WEEK	LA	PG	MAIN IPC
EP 1316798	A1	20030604	(200355)*	FR	8[1]	
WO 2004051257	A1	20040617	(200440)#	FR		
EP 1316798	B1	20041013	(200467)	FR		
AU 2002364803	A1	20040623	(200472)#	EN		
DE 60106466	E	20041118	(200476)	DE		
ES 2231412	T3	20050516	(200535)	ES		
DE 60106466	T2	20050602	(200537)	DE		
US 20050287062	A1	20051229	(200603)#	EN		

#### APPLICATION DETAILS:

PATENT NO	KIND	APPLICATION	DATE
EP 1316798 A1		EP 2001-403036	20011127

DE 60106466 E  
 DE 60106466 T2  
 DE 60106466 E  
 ES 2231412 T3  
 DE 60106466 T2  
 AU 2002364803 A1  
 WO 2004051257 A1  
 AU 2002364803 A1  
 US 20050287062 A1  
 US 20050287062 A1

DE 2001-60106466 20011127  
 DE 2001-60106466 20011127  
 EP 2001-403036 20011127  
 EP 2001-403036 20011127  
 EP 2001-403036 20011127  
 AU 2002-364803 20021129  
 WO 2002-FR4115 20021129  
 WO 2002-FR4115 20021129  
 WO 2002-FR4115 20021129  
 WO 2002-FR4115 20021129  
 US 2005-536853 20050527

## FILING DETAILS:

PATENT NO	KIND	PATENT NO
DE 60106466 E	Based on	EP 1316798 A
ES 2231412 T3	Based on	EP 1316798 A
DE 60106466 T2	Based on	EP 1316798 A
AU 2002364803 A1	Based on	WO 2004051257 A

PRIORITY APPLN. INFO: EP 2001-403036 20011127  
 WO 2002-FR4115 20021129  
 AU 2002-364803 20021129  
 US 2005-536853 20050527

## INT. PATENT CLASSIF.:

MAIN: G01N0030-48

IPC RECLASSIF.: B01J0020-10 [I,A]; B01J0020-10 [I,C]; B01J0020-28 [I,A]; B01J0020-28 [I,C]; B01J0020-281 [I,C]; B01J0020-283 [I,A]; B01J0020-291 [I,A]; C01B0033-00 [I,C]; C01B0033-12 [I,A]

ECLA: B01J0020-10B; B01J0020-28; B01J0020-283; B01J0020-291

ICO: L01J0220:20

USCLASS NCLM: 423/335.000

## BASIC ABSTRACT:

EP 1316798 A1 UPAB: 20060120

NOVELTY - Columns for flash chromatography or solid phase extraction (SPE) contain porous, spherical or semi-spherical silica gel (I) of 3-45 micron particle size and porosity 30-300 Angstrom.

USE - The columns are useful for the rapid, laboratory-scale purification, under low or medium pressure, of synthetic chemicals in pharmaceutical, cosmetic, agrochemical and biotechnological research.

ADVANTAGE - The columns pre-filled with (I) have a markedly superior separation and purification performance to conventional columns containing irregularly shaped porous silica gel, while retaining the same ease of use and a low working counter-pressure. TECHNOLOGY FOCUS:

CHEMICAL ENGINEERING - Preferred Columns: The columns contain 10 mg-1 kg of (I), and are in the form of tubes, syringe bodies or the like.

## EXTENSION ABSTRACT:

EXAMPLE - 50 g of spherical, porous silica gel of 25-40 micron particle size and porosity of 70 Angstrom was introduced into a 150 ml, 37 mm diameter syringe body including a porous sintered base. A second porous plate was placed above the silica bed, followed by stabilizing the bed by compression and vibration to give a column of length 85 mm. A product containing 1,6-diazido-3,4-dibenzoyloxy-2,5-dihydroxy-hexane as main component was injected into the column. Elution was carried out using an ethyl acetate (EA)/hexane (HX) gradient (100 % EA at time 0 and 5, 90 % EA/10 % HX at time 5.1 and 10, 80 % EA/20 % HX at

time 10.1 and 15.0, 70 % EA/30 % HX at time 15.1 and 25) at a flow rate of 35 ml per minute, with UV detection at 254 nm. The number of plateaux at the main peak was 684, the K' value was 10, the working pressure was 16 psi and the asymmetry of the main peak was 1.75. For comparison, the corresponding values using a column of irregularly shaped porous silica gel were 156 plateaux at the main peak, K' value 10.5, working pressure 43 psi and asymmetry of the main peak 5.88.

FILE SEGMENT: CPI; EPI  
 MANUAL CODE: CPI: B05-B02C; B11-B;  
 B11-C; C05-B02C; C11-B;  
 C11-C; J01-D01A; J04-B01C  
 EPI: S03-E09C5

L37 ANSWER 2 OF 2 WPIX COPYRIGHT 2009 THOMSON REUTERS on STN  
 ACCESSION NUMBER: 1999-266021 [23] WPIX  
 DOC. NO. CPI: C1999-078615 [23]  
 DOC. NO. NON-CPI: N1999-198332 [23]  
 TITLE: Separation agent comprises optically active polymer deposited on carrier which is stable to treatment with solvents  
 DERWENT CLASS: A11; A13; A89; B04; C07; J04; S03  
 INVENTOR: AZNAR P  
 PATENT ASSIGNEE: (AZNA-I) AZNAR P  
 COUNTRY COUNT: 23

## PATENT INFORMATION:

PATENT NO	KIND DATE	WEEK	LA	PG	MAIN IPC
EP 915335	A1 19990512 (199923)*	EN	7	[2]	

## APPLICATION DETAILS:

PATENT NO	KIND	APPLICATION	DATE
EP 915335 A1		EP 1997-402387	19971010

PRIORITY APPLN. INFO: EP 1997-402387 19971010  
 INT. PATENT CLASSIF.:  
 IPC RECLASSIF.: B01J0020-30 [I,C]; B01J0020-32 [I,A]; C07B0057-00 [I,A]; C07B0057-00 [I,C]  
 ECLA: B01J0020-32; C07B0057-00; G01N0030-48A

## BASIC ABSTRACT:

EP 915335 A1 UPAB: 20050521

NOVELTY - A separation agent comprises an optically active polymer deposited together with a small amount of polystyrene crystalline or any polymers containing aromatic rings on an organic or inorganic carrier and which is stable to the treatment with most solvents.

USE - The separation agent is used for the chromatographic separation of optically active compounds (claimed).

ADVANTAGE - The polystyrene crystalline addition ensures that high enantioselectivity is obtained in a pure form. The higher chemical stability allows the use of many different solvents, e.g. chlorinated solvents. This is particularly advantageous many racemates are difficult to dissolve at the concentrations required in preparative chromatography.

DESCRIPTION OF DRAWINGS - The chromatographic spectra shows the two retention times derived from the separation of racemic transtyrene oxide in a column. TECHNOLOGY FOCUS:

ORGANIC CHEMISTRY - Preferred Separation:



The separation agent polymer is a derivative of cellulose or amylose. The separation agent polymer derivatives of cellulose or amylose are carbamates. The separation agent has a silica carrier.

## EXTENSION ABSTRACT:

EXAMPLE - 5 g Cellulose was dispersed in 50 ml fresh distilled pyridine. 2 g 4-Dimethylaminopyridine was added and the suspension boiled for 72 hours. After cooling the mixture was poured into MeOH and the precipitate was filtered, washed with MeOH and dried. 3.4 g Of this material was suspended in 10 ml hexane/IPA, stirred and sonicated for 5 minutes. The mixture was poured in a reservoir for high pressure packing (80 ml) and filled up. The column was then packed at 650 bar. The column was pushed with 100 ml hexane/IPA and equilibrated for 4 minutes. This column (4.6 x 250 mm) was used for the resolution of racemic transtylbene oxide, using hexane/IPA 9/1 as the eluant at a rate of 0.5 ml/minute and being detected using a UV detector at 254 nm. There were two distinct peaks with retention times (Rt) of 11.067 and 16.393 respectively.

FILE SEGMENT:

CPI; EPI

MANUAL CODE:

CPI: A04-C02E; A10-E24; A12-L04A; B04-C02A; B04-C03B;  
B05-B02C; B11-B; C04-C02A; C04-C03B; C05-B02C; C11-B;  
J04-B01C  
EPI: S03-E09C

=&gt; D L40 1-21 IFULL

L40 ANSWER 1 OF 21 WPIX COPYRIGHT 2009 THOMSON REUTERS on STN  
ACCESSION NUMBER: 2005-221987 [23] WPIX  
CROSS REFERENCE: 2004-080414  
DOC. NO. CPI: C2005-071044 [23]  
DOC. NO. NON-CPI: N2005-182971 [23]  
TITLE: Separation of enantiomeric isomers by simulated  
moving bed chromatography comprises  
bringing the enantiomeric isomers into contact with a  
filler comprising a porous carrier  
DERWENT CLASS: A11; A89; B07; J01; J04; S03  
INVENTOR: MURAZUMI K; OHNISHI A  
PATENT ASSIGNEE: (DAIL-C) DAICEL CHEM IND LTD  
COUNTRY COUNT: 1

## PATENT INFORMATION:

PATENT NO	KIND	DATE	WEEK	LA	PG	MAIN IPC
US 20050054878	A1	20050310	(200523)*	EN	19[12]	
US 7306734	B2	20071211	(200781)	EN		

## APPLICATION DETAILS:

PATENT NO	KIND	APPLICATION	DATE
US 20050054878	A1 Div Ex	US 2002-279738	20021024
US 20050054878	A1	US 2004-943436	20040917

PRIORITY APPLN. INFO: JP 2002-307820 20021023  
JP 2002-110542 20020412

## INT. PATENT CLASSIF.:

MAIN: C07C0051-573  
IPC ORIGINAL: B01D0015-08 [I,A]; B01D0015-08 [I,C]  
IPC RECLASSIF.: B01D0015-10 [I,C]; B01D0015-18 [I,A]; B01D0015-20

[I,A]; B01D0015-22 [N,A]; B01D0015-26 [I,C];  
B01D0015-38 [I,A]; B01J0020-30 [I,C];  
B01J0020-32 [I,A]; G01N0030-00 [N,C];  
G01N0030-56 [N,A]  
ECLA: B01D0015-18R2; B01D0015-20P; B01D0015-38B;  
B01J0020-32; G01N0030-48A  
ICO: L01D0015:22; L01D0015:38B+S01N30/56A1; S01N0030:56A1  
USCLASS NCLM: 562/401.000  
NCLS: 530/416.000

## BASIC ABSTRACT:

US 20050054878 A1 UPAB: 20050708

NOVELTY - Separation of enantiomeric isomers by simulated moving bed chromatography comprises bringing the enantiomeric isomers into contact with a filler comprising a porous carrier (carrying a polysaccharide derivative) and having a TS (tetrakis(trimethylsilyl)silane) coefficient of 0.55-1.20.

DETAILED DESCRIPTION - Separation of enantiomeric isomers by simulated moving bed chromatography comprises bringing the enantiomeric isomers into contact with a filler comprising a porous carrier (carrying a polysaccharide derivative) and having a TS (tetrakis(trimethylsilyl)silane) coefficient of 0.55-1.20, where the TS coefficient is determined by a formula (TS coefficient =  $(V_c - t(TS) - t(blank)) \times FR / (TS - t(blank)) \times FR$ ) and obtained by using an enantiomeric isomer separation column for simulated moving bed chromatography in which the filler is fitted in a column tube by a slurry filling method and where  $V_c$  (cm<sup>3</sup>) is a column volume; FR (ml/min) is a flow velocity;  $t(TS)$  (min) is an elution time for tetrakis (trimethylsilyl) silane; and  $t(blank)$  (min) is an elution time for TS in the state where the column is not connected.

USE - The invention deals with the separation of enantiomeric isomers.

ADVANTAGE - The process separates the enantiomeric mixtures efficiently. The TS coefficient significantly improve the accuracy of confirmation of separation ability as compared with the conventional confirmation means based on the carrying amount of polysaccharide derivative. The polysaccharide has a high regularity of binding. TECHNOLOGY FOCUS:

ORGANIC CHEMISTRY - Preferred Process: The TS coefficient is 0.55-1.0; or greater than 1.0 but not greater than 1.20. The porous carrier is silica gel having a particle diameter of 1-300 microns and an average pore diameter of 200-8,000 Angstroms. The polysaccharide derivative (27-35 % by mass) is a cellulose ester derivative, a cellulose carbamate derivative, an amylose ester derivative or preferably an amylose carbamate derivative (amylose tris(3,5-dimethylphenyl carbamate)) and is carried on the porous carrier in an amount of from 23-40 % (preferably 35 %) by mass. The method separates 0.1-4 kg of enantiomeric isomers per kg of filler per day. The method additionally comprises deposition of 2-6 coatings of the polysaccharide derivative onto the porous carrier. The simulated moving bed chromatography is performed in a column having the filler packed. The column has a ratio (0.01-100 (preferably 0.01-30) column length (L) to column inner diameter (D).

## EXTENSION ABSTRACT:

EXAMPLE - Amylose (100 g) and 3,5-dimethylphenyl isocyanate (850 g) were heated under a nitrogen atmosphere and stirred in dry pyridine (4 liters) at 100 degrees C for 60 hours and the reaction mixture was poured into methanol (60 liters). The solids deposited were filtered and washed with methanol and dried under vacuum at 60 degrees C for 15 hours and yellowish white powdery solids of amylose tris(3,5-dimethylphenyl carbamate) (335 g, yield 90%) was obtained. Amylose tris(3,5-dimethylphenyl carbamate) (87.5 g) was dissolved in an ethyl acetate (747 ml). In a planetary stirrer type mixer surface inactivation-treated silica gel

(162.5 g) was charged and uniformly coated with a 1/4 portion of the polymer dope. After completion of the coating, the solvent was distilled off under heating and under reduced pressure conditions. The procedure was repeated several times to obtain amylose tris(3,5-dimethylphenyl carbamate)-carrying enantiomeric isomer.

FILE SEGMENT: CPI; EPI  
 MANUAL CODE: CPI: A03-A01; A12-L04A; B04-C02; B05-B02C;  
 B11-B; J01-D01A; J04-B01C  
 EPI: S03-E09C

L40 ANSWER 2 OF 21 WPIX COPYRIGHT 2009 THOMSON REUTERS on STN  
 ACCESSION NUMBER: 2004-399133 [37] WPIX  
 DOC. NO. CPI: C2004-149388 [37]  
 TITLE: Production of mesoporous silica  
 bead liquid chromatography packing  
 involves hydrolyzing compound containing silicon  
 followed by mixing with surfactant  
 DERWENT CLASS: A89; E11; E36; G02  
 INVENTOR: MODREK-NAJAFABADI B  
 PATENT ASSIGNEE: (MODR-I) MODREK-NAJAFABADI B; (VARI-C) VARIAN INC  
 COUNTRY COUNT: 31

## PATENT INFORMATION:

PATENT NO	KIND	DATE	WEEK	LA	PG	MAIN IPC
US 20040091411	A1	20040513	(200437)*	EN	15	[3]
WO 2004043861	A2	20040527	(200437)	EN		
AU 2003286869	A1	20040603	(200470)	EN		
EP 1562857	A2	20050817	(200554)	EN		
JP 2006505402	W	20060216	(200614)	JA	22	

## APPLICATION DETAILS:

PATENT NO	KIND	APPLICATION	DATE
US 20040091411	A1	US 2002-290863	20021108
AU 2003286869	A1	AU 2003-286869	20031031
EP 1562857	A2	EP 2003-778085	20031031
WO 2004043861	A2	WO 2003-US34972	20031031
EP 1562857	A2	WO 2003-US34972	20031031
JP 2006505402	W	WO 2003-US34972	20031031
JP 2006505402	W	JP 2004-551681	20031031

## FILING DETAILS:

PATENT NO	KIND	PATENT NO
AU 2003286869	A1	Based on WO 2004043861 A
EP 1562857	A2	Based on WO 2004043861 A
JP 2006505402	W	Based on WO 2004043861 A

PRIORITY APPLN. INFO: US 2002-290863 20021108

## INT. PATENT CLASSIF.:

IPC ORIGINAL: B01J0020-281 [I,A]; B01J0020-281  
 [I,C]; B01J0020-283 [I,A]; C01B0033-00  
 [I,C]; C01B0033-12 [I,A]; G01N0030-00 [I,C]  
 ; G01N0030-60 [I,A]; G01N0030-88 [I,A]

## IPC RECLASSIF.:

B01J0020-10 [I,A]; B01J0020-10  
 [I,C]; B01J0020-28 [I,A];

B01J0020-28 [I,C]; B01J0020-30  
[I,A]; B01J0020-30 [I,C]  
ECLA: B01J0020-10B; B01J0020-28; B01J0020-30; G01N0030-48A  
USCLASS NCLM: 423/338.000  
NCLS: 423/335.000  
JAP. PATENT CLASSIF.:  
MAIN/SEC.: C01B0033-12 Z; G01N0030-48 G; G01N0030-48 K;  
G01N0030-60 A  
FTERM CLASSIF.: 2G063; 4G072; 4G072/AA25; 4G072/BB07; 4G072/GG01;  
4G072/GG03; 4G072/HH30; 4G072/JJ47; 4G072/KK01;  
4G072/LL11; 4G072/MM22; 4G072/PP01; 4G072/PP05;  
4G072/RR05; 4G072/TT05; 4G072/TT08; 4G072/TT09;  
4G072/UU13

## BASIC ABSTRACT:

US 20040091411 A1 UPAB: 20060121

NOVELTY - Production of a mesoporous silica bead liquid chromatography packing involves hydrolyzing a compound containing silicon to form silica sol (S1); mixing (S1) with a dispersive medium containing at least one surfactant to form sol droplets (D1); transferring (D1) to a gelling medium at velocity of at least 3 m/second to form gelled product (G1); isolating (G1) from any non-gelled material to form an isolated product (I1); and calcining (I1).

DETAILED DESCRIPTION - Production of a mesoporous silica bead liquid chromatography (LC) packing (M1) involves

(1) acid-catalyzed hydrolyzing a compound containing silicon to form a silica sol (S1);

(2) mixing (S1) with a dispersive medium containing at least one surfactant to form sol droplets (D1);

(3) transferring (D1) to a gelling medium at a linear velocity of at least 3 m/second to form gelled product (G1);

(4) isolating (G1) from any non-gelled material to form an isolated product (I1); and

(5) calcining (I1).

An INDEPENDENT CLAIM is also included for an LC column comprising a durable support and (M1) in contact with the durable support.

USE - Producing mesoporous silica bead liquid chromatography (LC) packing (claimed).

ADVANTAGE - The method provides high purity, high surface area silica spherical beads having average diameter of 2-9 microns with high porosity and narrow pore size distribution used in LC columns such as high performance LC columns having good mechanical strength. The method provides superior separations properties of (M1). The pore diameter is large enough to facilitate functionalization of the beads while maintaining a high degree of mechanical stability and avoiding high back pressures. TECHNOLOGY FOCUS:

ORGANIC CHEMISTRY - Preferred Process: The hydrolysis is catalyzed by an acid. The transferring step (3) involves employing an apparatus consisting of an emulsion and a nozzle. The transferring is followed by mixing the gelling medium and the transferred (D1). (D1) are formed by mixing (S1) with dispersive medium containing surfactant (0.5%) and stirring the dispersive medium. The isolation involves isolating the gelled product from any non-gelled material by filtration, centrifugation or decanting; and washing (I1) with alcohol, water or organic solvents.

The calcination involves placing (I1) in a vacuum oven at ambient temperature; vacuum drying; placing (I1) in a furnace at ambient temperature; increasing the temperature over 24 hours and baking the isolated gel. The method further involves following calcining, adding water to (M1) and boiling it with stirring to form a hydrated product; separating the hydrated product from the water by filtration to form isolated hydrated product; and drying the product. The method further involves aging the

gelled product before isolation.

**Preferred Components:** The compound contains silicon (preferably alkoxy silane). The acid is selected from organic acid and/or mineral acids. The dispersive medium contains alcohol containing at least 8 carbon atoms. The gelation medium contains dispersive medium, a surfactant and a base. The base contains at least one organic base. (S1) is formed by mixing water at pH 0.7-2.0 with tetraethoxysilane (TEOS). The support comprises a tube having an inner diameter of 1-50 mm. The durable support is formed from stainless steel.

**POLYMERS - Preferred Components:** The surfactant is polyoxyethylene sorbitans, polyoxyethylene ethers, tri-block copolymers, alkyltrimethylammonium and/or surfactant containing octylphenol polymerized with ethylene oxide. The durable support is formed from a material selected from poly(etheretherketone) (PEEK).

**INORGANIC CHEMISTRY - Preferred Packing:** The packing comprises a surface area greater than 450 m<sup>2</sup>/g and an average pore diameter of 100 Angstrom. (M1) has an average pore size of 60-300 Angstrom. The pores have a uniform pore size. (M1) has a pore volume of greater than 1.2 ml/g. (M1) has a dimensions of 2-9 microns. The product of average pore diameter (Angstrom) value multiplied by the pore volume (in ml/g) value multiplied by the surface area (in m<sup>2</sup>/g) value of packing is greater than 55000.

#### EXTENSION ABSTRACT:

**EXAMPLE - Tetraethoxysilane** (720 ml) and deionized water (1000 ml) at pH 1.8 were mixed and stirred for 30 minutes at 20 degrees C in a reactor. After 50 minutes, mixture of decyl alcohol (4000 ml) and surfactant (5 ml) (sorbitan monooleate (3 parts by volume) and TWEEN 80 (RTM; surfactant)(1 part by volume)) were added and stirred for 10 minutes. While stirring, the mixture was transferred into the mixture of decyl alcohol (2250 ml), imidazole (90 g) and the surfactant (25 ml). The reactor was pressurized to 80 psi at a linear velocity of transfer 4.7 m/second and stirring was continued for 25 minutes. After 24 hours the mixture was heated at 65 degrees C for 5 hours. The formed gel was then filtered, dried at room temperature overnight, again heated to 165 degrees C and cooled to obtain silica (216 g). The silica was heated to 550 degrees C over 24 hours. The silica was calcined for 70 hours at this temperature and high performance liquid chromatography (HPLC) water (1 l) was added and boiled with stirring for 24 hours. The silica was filtered and dried at 75 degrees C to obtain mesoporous silica bead LC packing having surface area of 540 m<sup>2</sup>/g, pore size of 98 Angstrom and pore volume of 1.34 ml/g.

FILE SEGMENT:

CPI

MANUAL CODE:

CPI: A12-L04A; E31-P01; G02-A05E

L40 ANSWER 3 OF 21

WPIX COPYRIGHT 2009

THOMSON REUTERS on STN

ACCESSION NUMBER:

2003-743830 [70] WPIX

CROSS REFERENCE:

1998-386901; 1998-506688; 1999-009395; 1999-045150;  
1999-070314; 1999-105524; 1999-167458; 1999-190174;  
1999-277659; 1999-287986; 1999-313210; 1999-457857;  
2000-349558; 2000-375583; 2000-557659; 2000-678760;  
2001-071577; 2001-244716; 2001-381916; 2001-463931;  
2001-548990; 2001-611309; 2001-637908; 2001-648288;  
2002-055352; 2002-065549; 2002-082150; 2002-204872;  
2002-314786; 2002-424420; 2002-424729; 2002-566535;  
2002-689463; 2002-690602; 2003-017190; 2003-093162;  
2003-110192; 2003-196844; 2003-218954; 2003-310425;

2003-615241; 2003-706911; 2003-777165; 2004-040958;  
 2004-051045; 2004-106502; 2004-118797; 2004-118818  
 C2003-204231 [70]  
 TITLE: Separation of mixture of polynucleotides involves  
 applying mixture of polynucleotides to separation  
 medium having non-polar surfaces and eluting mixture  
 of polynucleotides  
 DERWENT CLASS: B04; D16; J01; J04  
 INVENTOR: GJERDE D T; TAYLOR P D  
 PATENT ASSIGNEE: (GJER-I) GJERDE D T; (TAYL-I) TAYLOR P D  
 COUNTRY COUNT: 1

## PATENT INFORMATION:

PATENT NO	KIND DATE	WEEK	LA	PG	MAIN IPC
US 20030075503	A1	20030424	(200370)*	EN	35 [24]

## APPLICATION DETAILS:

PATENT NO	KIND	APPLICATION	DATE
US 20030075503	A1	Provisional	US 1997-69313P 19971205
US 20030075503	A1	Provisional	US 1998-77998P 19980313
US 20030075503	A1	CIP of	US 1998-58337 19980410
US 20030075503	A1	Provisional	US 1998-89606P 19980617
US 20030075503	A1	Provisional	US 1998-103313P 19981006
US 20030075503	A1	Cont of	US 1998-183450 19981030
US 20030075503	A1	Cont of	US 2000-493734 20000128
US 20030075503	A1		US 2002-85691 20020227

## FILING DETAILS:

PATENT NO	KIND	PATENT NO
US 20030075503	A1	Cont of
US 20030075503	A1	Cont of
		US 6056877 A
		US 6372130 B

PRIORITY APPLN. INFO: US 2002-85691 20020227  
 US 1997-69313P 19971205  
 US 1998-77998P 19980313  
 US 1998-58337 19980410  
 US 1998-89606P 19980617  
 US 1998-103313P 19981006  
 US 1998-183450 19981030  
 US 2000-493734 20000128

## INT. PATENT CLASSIF.:

IPC RECLASSIF.: B01D0015-08 [I,A]; B01D0015-08 [I,C]  
 USCLASS NCLM: 210/635.000  
 NCLS: 210/502.100

## BASIC ABSTRACT:

US 20030075503 A1 UPAB: 20050601  
 NOVELTY - A mixture of polynucleotides having up to 1500 base pairs applied to a separation medium and eluted to separate the polynucleotides mixture, is new. The separation surfaces of the medium are coated with a hydrocarbon or non-polar hydrocarbon substituted polymer, or has substantially all polar groups reacted with a non-polar hydrocarbon or substituted hydrocarbon group. The surfaces of the medium are non-polar.

DETAILED DESCRIPTION - INDEPENDENT CLAIMS are also included for:

(1) a bead which comprises non-porous particle coated with a polymer and has average diameter of 0.5-100 micron and Mutation separation Factor of 0.1;

(2) treating the bead in order to improve the resolution of polynucleotides separated using the bead, which involves contacting a solution containing a multivalent cation binding agent with the bead and having temperature of 50-90degreesC;

(3) storing the bead which involves contacting the solution containing multivalent cation binding agent with the beads prior to storing the bead;

(4) silica gel monolith having non-polar interfacial separation surfaces, which is subjected to acid wash treatment in order to remove multivalent cation contaminant from surfaces;

(5) treating the monolith; and

(6) storing the monolith.

USE - For separating mixture of polynucleotides.

ADVANTAGE - The mixture of polynucleotides are separated efficiently using the non-porous beads. The column separation efficiency is preserved by storing the column separation media in the column containing a solution of multivalent cation binding agent.

DESCRIPTION OF DRAWINGS - The figure shows the schematic drawing of the cross-section of the reverse phase bead with silica core and polymer shielding.  
TECHNOLOGY FOCUS:

POLYMERS - Preferred Process: The mixture of polynucleotides is eluted with a mobile phase comprising counter ion agent and water-soluble organic solvent. The separation is by Matched Ion Polynucleotide Chromatography, capillary electrochromatography, thin layer chromatography or high-speed thin layer chromatography.

Preferred Properties: The medium has DNA Separation Factor of 0.05 and Mutation Separation Factor of 0.1. The nonporous beads and separation surfaces are substantially free from unreacted silanol groups.

ORGANIC CHEMISTRY - Preferred Solvent: The organic solvent is chosen from alcohol, nitrile, dimethylformamide, tetrahydrofuran, ester and/or ether. The counterion agent is chosen from lower alkyl primary-, secondary-, tertiary-amine, lower alkyl trialkylammonium salt and/or quaternary ammonium salt, preferably octylammonium acetate, octadimethyl ammonium acetate, decylammonium acetate, octadecylammonium acetate, pyridinium ammonium acetate, cyclohexylammonium acetate, diethylammonium acetate, propylethylammonium acetate, propyldiethyl ammonium acetate, butylethylammonium acetate, methylhexylammonium acetate, tetramethyl ammonium acetate, tetraethyl ammonium acetate, tetrapropyl ammonium acetate, tetrabutyl ammonium acetate, dimethyldiethyl ammonium acetate, triethylammonium acetate, tripropylammonium acetate, tributyl ammonium acetate and/or triethyl ammonium hexafluoroisopropyl alcohol. The counterion agent contains an anion chosen from acetate, carbonate, phosphate, sulfate, nitrate, propionate, formate, chloride and bromide.

INORGANIC CHEMISTRY - Preferred Particle: The nonporous particles are chosen from silica, silicon carbide, silicon nitride, titanium oxide, aluminum oxide, zirconium oxide, carbon, insoluble polysaccharide and diatomaceous earth, preferably silica reacted to make a reverse phase material.

Preferred Monolith: The monolith has the separation surface substrate groups end-capped with a non-polar hydrocarbon or substituted hydrocarbon group.

#### EXTENSION ABSTRACT:

EXAMPLE - Non-porous silica (in g) (200)  
was heated in a flask at 125degreesC and n-octadecyl dimethylsilane

was added. n-octadecyl methylchlorosilane (125), chloroform (10 ml), toluene (400 ml) and pyridine (65 ml) were charged in another flask. The liquid reagents were mixed and added to dry silica. The mixture was refluxed for 15 hours and cooled. Trimethyl chlorosilane (20 ml) and hexamethyl silane (6 ml) in toluene (20 ml) were added as capping reagent and the mixture was resuspended and refluxed for 6 hours. The mixture was cooled, and filtered and dried to obtain octadecyl modified, non-porous silica reverse phase material for column packing. The prepared material was packed in a column and used for separation of DNA restriction fragments. Double stranded DNA molecules were separated with high resolution.

FILE SEGMENT: CPI  
 MANUAL CODE: CPI: B04-C03; B04-E01; B11-C08D2; D05-H12;  
 J01-D01A; J04-B01C

L40 ANSWER 4 OF 21 WPIX COPYRIGHT 2009 THOMSON REUTERS on STN  
 ACCESSION NUMBER: 2003-256611 [25] WPIX  
 DOC. NO. CPI: C2003-066652 [25]  
 TITLE: Porous inorganic/organic hybrid monolith  
 material for chromatographic separations  
 device and chromatographic columns  
 comprises chromatographically-enhancing  
 pore geometry  
 DERWENT CLASS: A26; A97; E11; J01; J04; P73  
 INVENTOR: DING J; IRANETA P C; KELE M; O'GARA J E; WALTER T H;  
 OGARA J E  
 PATENT ASSIGNEE: (WATE-N) WATERS INVESTMENTS LTD  
 COUNTRY COUNT: 99

## PATENT INFORMATION:

PATENT NO	KIND	DATE	WEEK	LA	PG	MAIN IPC
WO 2003014450	A1	20030220	(200325)*	EN	99[0]	
US 20030150811	A1	20030814	(200355)	EN		
EP 1417366	A1	20040512	(200431)	EN		
AU 2002324647	A1	20030224	(200461)	EN		
JP 2004538468	W	20041224	(200502)	JA	178	
US 20070135304	A1	20070614	(200741)	EN		
US 7250214	B2	20070731	(200751)	EN		
JP 2009053198	A	20090312	(200920)	JA	79	
JP 4216716	B2	20090128	(200920)	JA	60	

## APPLICATION DETAILS:

PATENT NO	KIND	APPLICATION	DATE
WO 2003014450	A1	WO 2002-US25193	20020808
US 20030150811	A1 Provisional	US 2001-311445P	20010809
US 20070135304	A1 Provisional	US 2001-311445P	20010809
US 7250214	B2 Provisional	US 2001-311445P	20010809
AU 2002324647	A1	AU 2002-324647	20020808
EP 1417366	A1	EP 2002-759304	20020808
US 20030150811	A1	US 2002-216674	20020808
US 20070135304	A1 Div Ex	US 2002-216674	20020808
US 7250214	B2	US 2002-216674	20020808
EP 1417366	A1	WO 2002-US25193	20020808
JP 2004538468	W	WO 2002-US25193	20020808
JP 2004538468	W	JP 2003-519572	20020808



US 20070135304 A1  
 JP 4216716 B2 PCT Application  
 JP 2009053198 A Div Ex  
 JP 4216716 B2  
 JP 2009053198 A

US 2006-644279 20061221  
 WO 2002-US25193 20020808  
 JP 2003-519572 20020808  
 JP 2003-519572 20020808  
 JP 2008-236842 20080916

## FILING DETAILS:

PATENT NO	KIND	PATENT NO
EP 1417366	A1 Based on	WO 2003014450 A
AU 2002324647	A1 Based on	WO 2003014450 A
JP 2004538468	W Based on	WO 2003014450 A
JP 4216716	B2 Previous Publ	JP 2004538468 W
JP 4216716	B2 Based on	WO 2003014450 A

PRIORITY APPLN. INFO: US 2001-311445P 20010809  
 US 2002-216674 20020808  
 US 2006-644279 20061221  
 US 2001-311445P 20010809

## INT. PATENT CLASSIF.:

MAIN: D04H0001-00; G01N0030-48  
 SECONDARY: D04H0013-00; D04H0003-00; D04H0005-00  
 IPC ORIGINAL: B01J0020-22 [I,C]; B01J0020-26 [I,A];  
 B01J0020-281 [I,C]; B32B0015-02 [I,A];  
 B32B0015-02 [I,C]; G01N0030-00 [N,A]; G01N0030-00  
 [I,C]; G01N0030-00 [I,C]; G01N0030-00 [N,C];  
 G01N0030-26 [N,A]; G01N0030-54 [N,A]; G01N0030-56  
 [N,A]; G01N0030-74 [N,A]; G01N0030-88 [I,A];  
 G01N0030-90 [I,A]  
 IPC RECLASSIF.: B01D0015-08 [I,A]; B01D0015-08 [I,C]; B01J0020-22  
 [I,C]; B01J0020-26 [I,A];  
 B01J0020-28 [I,A]; B01J0020-28  
 [I,C]; B01J0020-281 [I,A];  
 B01J0020-281 [I,C]; B01J0020-286  
 [I,A]; B01J0020-30 [I,C]; B01J0020-32 [I,A]  
 ; B01J0021-00 [I,C]; B01J0021-04 [I,A]; C02F0001-28  
 [I,A]; C02F0001-28 [I,C]; D04H0001-42 [I,A];  
 D04H0001-42 [I,C]; G01N0030-00 [I,A]; G01N0030-00  
 [I,C]; G01N0030-26 [I,A]; G01N0030-54 [I,A];  
 G01N0030-56 [I,A]; G01N0030-74 [I,A]; G01N0030-88  
 [I,A]; G01N0030-90 [I,A]  
 ECLA: B01J0020-28; B01J0020-286; B01J0020-32; D04H0001-42  
 ICO: L01J0220:20  
 USCLASS NCLM: 210/656.000; 502/402.000  
 NCL: 502/402.000; 502/527.180

## ECLA:

## ICO:

## USCLASS NCLM:

## NCL:

## JAP. PATENT CLASSIF.:

MAIN/SEC.: B01J0020-22 D; G01N0030-00 J; G01N0030-26 A;  
 G01N0030-48 C; G01N0030-48 D; G01N0030-48 G;  
 G01N0030-48 L; G01N0030-54 F; G01N0030-54 K;  
 G01N0030-56 A; G01N0030-74 E; G01N0030-88 101 C;  
 G01N0030-88 101 D; G01N0030-88 101 E; G01N0030-88 101  
 H; G01N0030-88 101 K; G01N0030-88 101 L; G01N0030-88  
 201 G; G01N0030-88 201 X; G01N0030-88 C; G01N0030-90  
 G01N0030-88 201 G  
 MAIN: B01J0020-22 D; G01N0030-88 101 C; G01N0030-88 101 D;  
 SECONDARY: G01N0030-88 101 E; G01N0030-88 101 H; G01N0030-88 101  
 L; G01N0030-88 201 X; G01N0030-88 C; G01N0030-90  
 ADDITIONAL: G01N0030-00 J; G01N0030-26 A; G01N0030-54 F;  
 G01N0030-54 K; G01N0030-56 A; G01N0030-74 E

## FTERM CLASSIF.:

2G063; 4G066; 4G066/AA20.C; 4G066/AA22.C;  
4G066/AA23.C; 4G066/AA72.C; 4G066/AB18.B;  
4G066/AB18.D; 4G066/BA07; 4G066/BA23; 4G066/BA25;  
4G066/BA26; 4G066/EA01; 4G066/FA05; 4G066/FA21

## BASIC ABSTRACT:

WO 2003014450 A1 UPAB: 20090401

NOVELTY - A porous inorganic/organic hybrid monolith material comprises a chromatographically-enhancing pore geometry.

DETAILED DESCRIPTION - INDEPENDENT CLAIMS are included for:

(a) a method of preparation of a porous inorganic/organic hybrid monolith material comprising forming porous inorganic/organic hybrid particles; modifying the pore structure of the porous hybrid particles, and coalescing the porous hybrid particles to form a monolith material; and

(b) a chromatographic column having improved lifetime comprising a column having a cylindrical interior for accepting a monolith material of porous inorganic/organic hybrid material; and a chromatographic bed comprising a porous inorganic/organic hybrid monolith material.

USE - For chromatographic separations device, e.g. chromatographic columns, thin layer plates, filtration membranes, sample cleanup devices, and microtiter plates (claimed).

ADVANTAGE - The hybrid materials have low micropore surface area, thus giving chromatographic enhancements including high separation efficiency and good mass transfer properties (as evidenced by e.g., reduced band spreading and good peak shape). TECHNOLOGY FOCUS:

ORGANIC CHEMISTRY - Preferred Properties: Pores of a diameter of less than 34 Angstrom contribute less than 110 m<sup>2</sup>/g to less than 50 m<sup>2</sup>/g to the specific surface area of the material.

The hybrid particles have a specific surface area of 50-800 m<sup>2</sup>/g, preferably 75-600 m<sup>2</sup>/g, or more preferably 100-350 m<sup>2</sup>/g. They have specific pore volumes of 0.25-1.5 cm<sup>3</sup>/g, preferably 0.4-1.2 cm<sup>3</sup>/g. They have a micropore surface area of less than 110 m<sup>2</sup>/g, preferably less than 105 m<sup>2</sup>/g, or more preferably 50 m<sup>2</sup>/g. They have an average pore diameter of 50-500 Angstrom, preferably 100-300 Angstrom.

Preferred Components: The porous inorganic/organic hybrid monolith material comprises coalesced porous inorganic/organic hybrid particles having a chromatographically-enhancing pore geometry, where the particles have been surface modified by a surface modifier consisting of organic group surface modifier, silanol group surface modifier, and/or polymeric coating surface modifier.

The hybrid particles have been surface modified with a surface modifier of formula  $Za(R')bSiR$ .

Z = Cl, Br, I, 1-5C alkoxy, dialkylamino or trifluoromethanesulfonate;

R = 1-6C alkyl;

R = functionalizing group;

a and b = 0-3, provided that a+b is 3.

The surface modifier is octyltrichlorosilane, octadecyltrichlorosilane, octyldimethylchlorosilane, or octadecyldimethylchlorosilane, preferably octyltrichlorosilane or octadecyltrichlorosilane.

The hybrid monolith material is of formula  $SiO_2 / (R_2pR_4qSiOt)_n$  or  $SiO_2 / (R_6(R_2rSiOt)_m)_n$ .

R2 and R4 = 1-18 aliphatic, styryl, vinyl, propanol, or aromatic moieties;

R6 = optionally substituted 1-18C alkylene, alkenylene, alkenylene or arylene moiety bridging two or more silicon atoms;  
n = 0.03-1;

p and q = 0, 1, or 2, provided that p+q is 1 or 2;

when  $p+q$  is 1,  $t = 1.5$ ;  
 when  $p+q$  is 2,  $t = 1$ ;  
 $r = 0$  or 1, provided that when  $r$  is 0,  $t$  is 1.5 and when  $r$  is 1,  $t$  is 1.

The surfactant or combination of surfactants are Triton X-45(TM), sodium dodecylsulfate, and/or tris(hydroxymethyl)aminomethane lauryl sulfate.

The suspension further comprises a porogen, which is toluene.

The tetraalkoxysilane is of formula  $\text{Si}(\text{OR})_4$  and he organoalkoxysilane of formula  $\text{R}_2'\text{Si}(\text{OR}')_3$  or  $\text{R}_6'(\text{SiOR}')_3\text{m}$ .

$\text{R}_1 = 1\text{-}3\text{C}$  alkyl moiety;

$\text{R}_2' = 1\text{-}18\text{C}$  aliphatic, styryl, vinyl, propanol or aromatic moiety;

$\text{R}_1' = 1\text{-}4\text{C}$  alkyl moiety;

$\text{R}_6' = 1\text{-}18\text{C}$  alkylene, alkenylene, alkynylene or arylene moiety bridging two or more silicon atoms; and

$m = \text{at least } 2$

POLYMERS - Preferred Components: The polymer is Sylgard(TM).

Preferred Method: The porous hybrid particles are prepared by prepolymerizing one or more organoalkoxysilanes and/or tetraalkoxysilane to produce a polyorganoalkoxysiloxane, and preparing an aqueous suspension of the polyorganoalkoxysiloxane, and gelling in the presence of a base catalyst to produce the porous hybrid particles.

The pore structure of the porous hybrid particles is modified by further including a surfactant or combination of different surfactants in the suspension, and by subjecting the porous hybrid particles to hydrothermal treatment.

The prepolymerization step comprises hydrolyzing and condensing a mixture of one or more organoalkoxysilanes and a tetraalkoxysilane in the presence of an acid catalyst to produce the polyalkyloxysiloxane.

The organo(tri)alkoxysilane and tetraalkoxysilane is 0.5:1-0.2:1.

INORGANIC CHEMISTRY - Preferred Components: The inorganic portion of the hybrid monolith material is alumina, silica, titanium, zirconium oxides, or ceramics materials.

The base catalyst is ammonium hydroxide.

#### EXTENSION ABSTRACT:

DEFINITIONS - Preferred Definitions: -  $\text{R}' = \text{methyl, ethyl, propyl, isopropyl, butyl, t-butyl, sec-butyl, pentyl, isopentyl, hexyl, or cyclohexyl}$ ; -  $\text{R} = \text{most preferably } 1\text{-}20\text{C alkyl}$ ; -  $n = \text{preferably } 0.20\text{-}0.5$ .

EXAMPLE - A mixture of surfactant (Triton X-45(TM) or Triton X-100(TM)), ethanol, deionized water was heated at 55 degreesC for 0.5 hour, resulting in a white liquid. Under rapid agitation, a solution of toluene in polyorganoalkoxysiloxane was added into the ethanol/water/Triton mixture, and emulsified in the aqueous phase. 30% ammonium hydroxide was added into the emulsion to gel the emulsion beads. Suspended in the solution, the gelled product was transferred to a flask and stirred at 55 degreesC for 16 hours. The spherical, porous, hybrid inorganic/organic particles were collected on 0.5 microns filtration paper and washed successively with water and methanol. The products were then dried in a vacuum oven at 80 degreesC overnight.

FILE SEGMENT:	CPI; GMPI
MANUAL CODE:	CPI: A12-L04A; E05-E; E05-E02D; E05-E03; E31-P03; E34-C02; E35-K02; E35-L; J01-D01A; J04-B01C

L40 ANSWER 5 OF 21 WPIX COPYRIGHT 2009 THOMSON REUTERS on STN  
 ACCESSION NUMBER: 2002-732865 [79] WPIX  
 DOC. NO. CPI: C2002-207441 [79]  
 TITLE: New process for the preparation of maytansinol useful  
 as cell-binding agent maytansinol complex comprises  
 separation of maytansinol by normal phase high  
 performance liquid chromatography on e.g.  
 chemically modified silica stationary phase  
 DERWENT CLASS: B02  
 INVENTOR: TERFLOTH G J; TERFLOTH J  
 PATENT ASSIGNEE: (SMIK-C) SMITHKLINE BEECHAM CORP; (TERF-I) TERFLOTH G  
 J  
 COUNTRY COUNT: 99

## PATENT INFORMATION:

PATENT NO	KIND	DATE	WEEK	LA	PG	MAIN IPC
WO 2002074775	A1	20020926	(200279)*	EN	17[11]	
<--						
US 20020156274	A1	20021024	(200279)	EN		
<--						
EP 1373273	A1	20040102	(200409)	EN		
AU 2002257030	A1	20021003	(200432)	EN		
<--						
JP 2004526734	W	20040902	(200457)	JA	27	
US 20050113571	A1	20050526	(200535)	EN		
EP 1373273	B1	20051005	(200569)	EN		
DE 60206477	E	20060216	(200618)	DE		
ES 2248550	T3	20060316	(200622)	ES		
DE 60206477	T2	20060622	(200643)	DE		

## APPLICATION DETAILS:

PATENT NO	KIND	APPLICATION	DATE
WO 2002074775 A1		WO 2002-US7424	20020312
US 20020156274 A1	Provisional	US 2001-276792P	20010316
US 20050113571 A1	Provisional	US 2001-276792P	20010316
US 20020156274 A1		US 2002-95927	20020311
US 20050113571 A1	Cont of	US 2002-95927	20020311
AU 2002257030 A1		AU 2002-257030	20020312
DE 60206477 E		DE 2002-606477	20020312
EP 1373273 A1		EP 2002-726608	20020312
EP 1373273 B1		EP 2002-726608	20020312
DE 60206477 E		EP 2002-726608	20020312
ES 2248550 T3		EP 2002-726608	20020312
JP 2004526734 W		JP 2002-573784	20020312
EP 1373273 A1		WO 2002-US7424	20020312
JP 2004526734 W		WO 2002-US7424	20020312
EP 1373273 B1		WO 2002-US7424	20020312
DE 60206477 E		WO 2002-US7424	20020312
US 20050113571 A1		US 2004-146	20041130
DE 60206477 T2		DE 2002-606477	20020312
DE 60206477 T2		EP 2002-726608	20020312
DE 60206477 T2		WO 2002-US7424	20020312

## FILING DETAILS:

PATENT NO	KIND		PATENT NO	
DE 60206477	E	Based on	EP 1373273	A
ES 2248550	T3	Based on	EP 1373273	A
EP 1373273	A1	Based on	WO 2002074775	A
AU 2002257030	A1	Based on	WO 2002074775	A
JP 2004526734	W	Based on	WO 2002074775	A
EP 1373273	B1	Based on	WO 2002074775	A
DE 60206477	E	Based on	WO 2002074775	A
DE 60206477	T2	Based on	EP 1373273	A
DE 60206477	T2	Based on	WO 2002074775	A

PRIORITY APPLN. INFO: US 2001-276792P 20010316  
US 2002-95927 20020311  
US 2004-146 20041130

INT. PATENT CLASSIF.:  
MAIN: C07D0487-12; C07D0491-04; C07D0498-18  
SECONDARY: B01J0020-10  
IPC ORIGINAL: A61K0031-53 [I,A]; A61K0031-53 [I,A]; A61K0031-53 [I,C]; C07D0487-00 [I,C]; C07D0487-00 [I,C]; C07D0487-12 [I,A]; C07D0487-12 [I,A]  
IPC RECLASSIF.: B01J0020-10 [I,A]; B01J0020-10 [I,C]; B01J0020-281 [I,A]; B01J0020-281 [I,C]; C07D0498-00 [I,C]; C07D0498-18 [I,A]  
ECLA: C07D0498-18+303B+265B+225B  
USCLASS NCLM: 540/462.000  
NCLS: 210/656.000  
JAP. PATENT CLASSIF.:  
MAIN/SEC.: B01J0020-10 A; C07D0498-18 311  
FTERM CLASSIF.: 4C072; 4G066; 4C072/AA03; 4G066/AA20.B; 4G066/AA22.B; 4G066/AA23.B; 4G066/BA20; 4G066/BA23; 4G066/BA25; 4G066/BA26; 4G066/BA33; 4G066/BA38; 4C072/BB04; 4C072/BB06; 4G066/CA27; 4G066/CA33; 4C072/CC02; 4C072/CC12; 4G066/EA01; 4C072/EE06; 4C072/FF15; 4C072/GG07; 4C072/JJ05; 4C072/UU08

BASIC ABSTRACT:  
WO 2002074775 A1 UPAB: 20080523  
NOVELTY - New process for the preparation of maytansinol from a mixture containing unreduced and over-reduced maytansinoids comprises separation of maytansinol by normal-phase high performance liquid chromatography (HPLC) on a silica, alumina, zirconia, titanium dioxide or chemically modified silica stationary phase.  
USE - In the preparation of maytansinol and a cell-binding agent (e.g. antibody) maytansinoid complex (all claimed).  
ADVANTAGE - The process is economical and gives a large yield of the product. The process has variability in column bed efficiency and has a constant product quality irrespective of the operator. The process is reproducible and sealable. TECHNOLOGY FOCUS:  
INORGANIC CHEMISTRY - Preferred Process: The stationary phase is eluted with a halogenated hydrocarbon: aliphatic ester:alkanol mobile phase.  
The preparation involves separating maytansinol by HPLC on an angular particle porous amorphous silica gel stationary phase having either a median pore diameter (50 - 70) Angstrom, a pore volume (0.8 - 1.2) ml/g, a surface area (500 - 600) m<sup>2</sup>/g, a packed density (0.5 g/ml), less than 10% loss on drying, a 5% aqueous slurry pH 4 - 5.5, sodium content (less than 60 ppm), aluminum content (less than 100 ppm), iron content (less than 80 ppm), calcium content (less than 80 ppm), sulfate

content (less than 25 ppm) and chloride content (less than 25 ppm) eluted with a mobile phase of 50% dichloromethane : 39.3% ethyl acetate : 10.7% 2-propanol or a median pore diameter 60 Angstrom , a pore volume (0.9 ml/g), a surface area (700 m<sup>2</sup>/g), a bulk density (0.4 g/ml), a 15% aqueous slurry pH (6.5 - 7.5), iron content (less than 0.02)% and chloride content (less than 0.02%) eluted with a mobile phase of 30% dichloromethane:55% ethyl acetate:15% 2-propanol.

Preferred Components: The stationary phase is silica (preferably Silicycle IMPAQ (RTM) or Merck LICHROSPIER Si 60 (RTM)).

The stationary phase has a particle size of 5, 10, 20, 40, 80 or 150 microns.

#### EXTENSION ABSTRACT:

EXAMPLE - Chromatography was carried out using a Varex (RTM) preparative HPLC system with pump, feed pump and variable wavelength UV detector. A column was packed with IMPAQ (RTM; silica gel) (approximately 1 kg), porous amorphous silica gel stationary phase. The silica gel stationary phase had a pore diameter of 50 - 70 Angstrom , a pore volume of 0.8 - 1.2 ml/g, a surface area of 500 - 600 m<sup>2</sup>/g, a packed density of 0.5 g/ml, less than 10% loss on drying for 20 minutes at 205degreesC, a 5% aqueous slurry pH of 4.0 - 5.5, sodium content of less than 60 ppm, aluminum content of less than 100 ppm, iron content of less than 80 ppm, calcium content of less than 80 ppm, sulfate content of less than 25 ppm and chloride content of less than 25 ppm and a particle size of 10 microns. - A mobile phase of methylene chloride:ethyl acetate:2-propanol (50:39.3:10.7) at a flow rate of 500 ml/minutes was used. Detection was at 280 nm. The column was equilibrated for at least 10 minutes before each run. - Impure maytansinol (10.8 g) prepared by lithium trimethoxyaluminum hydride reduction of ansamitocin P-3 was dissolved in mobile phase (217 ml) to give a concentration of 49.8 mg/ml. This sample solution was recirculated for 10 minutes through a column (packed with the same silica as the preparative column) prior to pumping onto the preparative column. - A small fraction (F1) was taken on the front of the maytansinol peak followed by a main fraction (F2) and a small fraction (F3) of the tail. F1 had a purity of 90.8%, F2 of 99.4% and F3 of 99.7%. The second and third fractions from the three runs were combined and the solvent removed using a rotary evaporator to give a residue. This residue was redissolved in ethyl acetate (200 ml) and the solvent removed again. The flask was pumped for 2 hours under high vacuum giving 9.3 g of purified maytansinol. Under identical conditions, impure maytansinol (3.9 g) was dissolved in 89 ml of the mobile phase. The entire sample was purified in one injection giving 3.1 g of maytansinol at a purity of 99.3% by area.

FILE SEGMENT:

CPI

MANUAL CODE:

CPI: B06-E05; B12-K04

L40 ANSWER 6 OF 21

WPIX COPYRIGHT 2009

THOMSON REUTERS on SIN

ACCESSION NUMBER:

2002-673309 [72] WPIX

CROSS REFERENCE:

2000-558126; 2004-708502; 2008-C16185

DOC. NO. CPI:

C2002-189658 [72]

DOC. NO. NON-CPI:

N2002-532276 [72]

TITLE:

Porous (in)organic hybrid material, e.g.  
for chromatographic columns,  
comprises porous (in)organic hybrid  
particles having chromatographically

-enhancing pore geometry  
 A26; A89; J01; P42; P73; S03  
 DERWENT CLASS: FISK R P; JIANG Z; O'GARA J; WALTER T H; WYNDHAM K D  
 INVENTOR: (FISK-I) FISK R P; (JIANG-I) JIANG Z; (OGAR-I) O'GARA  
 PATENT ASSIGNEE: J; (WALT-I) WALTER T H; (WATE-N) WATERS INVESTMENTS  
 LTD; (WYND-I) WYNDHAM K D  
 COUNTRY COUNT: 98

## PATENT INFORMATION:

PATENT NO	KIND	DATE	WEEK	LA	PG	MAIN IPC
US 20020070168	A1	20020613	(200272)*	EN	21	[0]
<--						
WO 2003022392	A1	20030320	(200330)	EN		
US 6686035	B2	20040203	(200413)	EN		
EP 1450924	A1	20040901	(200457)	EN		
AU 2002362290	A1	20030324	(200461)	EN		
JP 2005502061	W	20050120	(200508)	JA	105	
JP 2009025315	A	20090205	(200916)	JA	54	

## APPLICATION DETAILS:

PATENT NO	KIND	APPLICATION	DATE
US 20020070168	A1 Cont of	US 1999-244795	19990205
US 20020070168	A1 CIP of	US 2001-858087	20010514
US 20020070168	A1	US 2001-924399	20010807
AU 2002362290	A1	AU 2002-362290	20020807
EP 1450924	A1	EP 2002-798083	20020807
WO 2003022392	A1	WO 2002-US25250	20020807
EP 1450924	A1	WO 2002-US25250	20020807
JP 2005502061	W	WO 2002-US25250	20020807
JP 2005502061	W	JP 2003-526515	20020807
JP 2009025315	A Div Ex	JP 2003-526515	20020807
JP 2009025315	A	JP 2008-231653	20080910

## FILING DETAILS:

PATENT NO	KIND	PATENT NO
EP 1450924	A1 Based on	WO 2003022392 A
AU 2002362290	A1 Based on	WO 2003022392 A
JP 2005502061	W Based on	WO 2003022392 A

PRIORITY APPLN. INFO: US 2001-924399 20010807  
 US 1999-244795 19990205  
 US 2001-858087 20010514

## INT. PATENT CLASSIF.:

MAIN: B01D0015-08; G01N0030-46  
 SECONDARY: B05D0007-00; B32B0005-16; G01N0030-48  
 IPC ORIGINAL: B01J0020-281 [I,A]; B01J0020-281  
 [I,C]; G01N0030-00 [I,C]; G01N0030-88 [I,A];  
 G01N0030-93 [I,A]  
 IPC RECLASSIF.: B01J0020-10 [I,A]; B01J0020-10  
 [I,A]; B01J0020-10 [I,C];  
 B01J0020-10 [I,C]; B01J0020-281  
 [I,A]; B01J0020-281 [I,C];  
 B01J0020-281 [I,C]; B01J0020-286  
 [I,A]; B01J0020-286 [I,A];

B01J0020-289 [I,A]; B01J0020-30 [I,C]; B01J0020-32 [I,A]; B01J0039-26 [I,A]; B01J0039-26 [I,C]; B01J0041-20 [I,A]; B01J0041-20 [I,C]; G01N0030-00 [I,A]; G01N0030-00 [I,C]; G01N0030-46 [I,A]; G01N0030-52 [N,A]; G01N0030-54 [I,A]; G01N0030-56 [I,A]; G01N0030-74 [I,A]; G01N0030-88 [I,A]; G01N0030-90 [I,A]; B01J0020-10B; B01J0020-286; B01J0020-289; B01J0020-32; B01J0039-26; B01J0041-20 L01J0020:32J; S01N0030:52C1  
 210/656.000; 428/304.400  
 USCLASS NCLM: 210/198.200; 422/261.000; 428/315.500; 428/316.600;  
 NCLS: 428/328.000; 428/329.000; 428/331.000; 428/403.000;  
 428/405.000

JAP. PATENT CLASSIF.:  
 MAIN/SEC.: B01J0020-02 D; B01J0020-10 C; G01N0030-00 J;  
 G01N0030-46 G; G01N0030-48 C; G01N0030-48 D;  
 G01N0030-48 L; G01N0030-54 F; G01N0030-54 K;  
 G01N0030-56 A; G01N0030-74 E; G01N0030-88 101 C;  
 G01N0030-88 101 D; G01N0030-88 101 L; G01N0030-88 201 G; G01N0030-88 201 X; G01N0030-88 C; G01N0030-90  
 MAIN: G01N0030-88 201 G  
 SECONDARY: B01J0020-22 D; G01N0030-88 101 L; G01N0030-88 201 X;  
 G01N0030-88 201 Y; G01N0030-93  
 FTERM CLASSIF.: 2G063; 4G066; 4G066/AA22.B; 4G066/AA22.C;  
 4G066/AB18.B; 4G066/AB18.D; 4G066/AD01.B;  
 4G066/AD13.B; 4G066/AD20.B; 4G066/AE20.D; 4G066/BA23;  
 4G066/BA26; 4G066/DA07; 4G066/EA01; 4G066/FA03;  
 4G066/FA07; 4G066/FA11; 4G066/FA21; 4G066/FA33;  
 4G066/FA38

## BASIC ABSTRACT:

US 20020070168 A1 UPAB: 20090313

NOVELTY - A porous (in)organic hybrid material comprises porous (in)organic hybrid particles having a chromatographically-enhancing pore geometry.

DETAILED DESCRIPTION - A porous (in)organic hybrid material comprises porous (in)organic hybrid particles having a chromatographically-enhancing pore geometry. The particles have been surface modified with a surface modifier having formula  $Za(R')bSi-R$ ,

Z = Cl, Br, I, 1-5C alkoxy, dialkylamino or trifluoromethanesulfonate;  
 a and b = 0-3;

R' = 1-6C straight, cyclic or branched alkyl;

and R = functionalizing group.

where a+b is 3.

INDEPENDENT CLAIMS are included for:

(a) A method of preparing porous (in)organic hybrid particles having chromatographically-enhancing pore geometry comprising forming porous (in)organic hybrid particles, modifying the pore structure of the particles, and surface modifying the particles; and

(b) A chromatographic column comprising a column having a cylindrical interior for accepting a packaging material, and packed chromatographic bed comprising porous (in)organic hybrid particles.

USE - For use in separation devices e.g. chromatographic columns, thin layer plates, filtration membranes, sample clean-up devices or microtiter plates (claimed).

ADVANTAGE - The inventive material has chromatographically-enhancing pore geometry, which offers more efficient chromatographic separations than that known in the art.

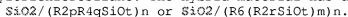
TECHNOLOGY FOCUS:

ORGANIC CHEMISTRY - Preferred Method: The porous



particles are prepared by prepolymerizing organoalkoxysilanes and tetraalkoxysilane to produce a polyorganoalkoxysiloxane, preparing an aqueous suspension of polyorganoalkoxysiloxane, and gelling in the presence of a base catalyst so as to produce the porous particles. The porous structure of the particles is modified by a surfactant in the suspension, and by subjecting the porous particles to hydrothermal treatment. The particles are surface modified by coating with a polymer or by organic and silanol group modifications, or by organic group modification or coating with polymer, or by silanol group modification and coating with polymer. The particles may also be surface modified via formation of organic covalent bond between the particles organic group and modifying reagent, or by combination of organic, silanol and coating with polymer, or solely by silanol group modification.

**Preferred Components:** The surface modifier has a formula  $Za(R')_bSi-R$ . The surface modifier is octyldimethylchlorosilane, octadecyldimethylchlorosilane, or preferably octyltrichlorosilane or octadecyltrichlorosilane. The hybrid material has a formula



The suspension further comprises a porogen, which can be toluene. The tetraalkoxysilane has the formula  $Si(OR)_4$ . The organoalkoxysilane has the formula  $R_2Si(OR)_3$  or  $R_6(Si(OR)_3)_m$ . The surfactant is Triton X-45 (RTM), sodium dococylsulfate, and/or tris(hydroxymethyl)aminomethane. The tetraalkoxysilane is preferably a tetramethoxysilane and tetraethoxysilane.

$R_1 = 1-4$  (preferably  $1-3$ ) C alkyl moiety (preferably Et or Me);  
 $R_2$  and  $R_4 = 1-18$  C aliphatic or aromatic moieties;  
 $R_2 =$  preferably Me, Et, Ph, vinyl, methacryloxypropyl, or styrylethyl;

$R_6 =$  optionally substituted  $1-18$  C alkylene, alkenylene, alkynylene or arylene moiety bridging at least 2 Si (preferably a bridging ethylene group);

$p$  and  $q = 0, 1, 2$ ;

$m =$  at least 2 (preferably 2);

$n = 0.03-1$  (preferably  $0.2-0.5$ ).

Provided that  $p+q$  can be 1 or 2, when  $p+q$  is 1, then  $t$  is 1.5 and when  $p+q$  is 2, then  $t$  is 1;  $r$  can be 0 or 1, when  $r$  is 0, then  $t$  is 1.5 and when  $r$  is 1 then  $t$  is 1.

**Preferred Parameters:** The particles have a specific surface area of 50-800, (preferably 100-200)  $m^2/g$ , specific pore volumes of 0.25-1.5 (preferably 0.4-1.2)  $cm^3/g$ , micropore surface area of less than 110 (preferably less than 50)  $m^2/g$ , and average pore diameter of 50-500 (preferably 100-300) Angstrom.

**INORGANIC CHEMISTRY - Preferred Materials:** The inorganic portion of hybrid material is alumina, titanium, or zirconium oxides, or ceramic materials, or preferably silica. The base catalyst is free of alkali or alkaline earth metal cations, preferably an ammonium hydroxide.

#### EXTENSION ABSTRACT:

**DEFINITIONS - Preferred Definitions:** -  $R' =$  Me, Et, propyl, isopropyl, butyl, *t*-butyl, sec-butyl, pentyl, isopentyl, hexyl, or cyclohexyl; -  $R =$  alkyl, alkenyl, alkynyl, aryl, cyano, amino, diol, nitro, ester, cation or anion exchange group, alkyl or aryl group containing embedded polar functionality, or  $1-30$  C (preferably  $1-20$  C) alkyl

**EXAMPLE -** A surfactant (type X-45), ethanol (530 ml), and deionized water (2310 ml) were heated at 55degreesC for 0.5 hour, resulting in a white liquid. Under rapid agitation, a solution of toluene (58 ml) in polyorganoalkoxysiloxane (479 g) was added into

the mixture and emulsified in the aqueous phase. The organoalkoxysilane was a methacryloxypropyl of formula  $H_2C=C(CH_3)CO_2C_3H_6Si(OCH_3)_3$ . Ammonium hydroxide (398 ml) was added into the emulsion to gel the emulsion beads. The gelled product was transferred to a flask and stirred at 55degreesC for 16 hour. The resulting spherical, porous, hybrid inorganic/organic particles were collected on 0.5 mum filtration paper and washed with water and methanol. The products were then dried in a vacuum oven at 80degreesC overnight. The molar ratio of organosiloxane/silicon dioxide in product was 0.1.

FILE SEGMENT: CPI; GMPI; EPI  
 MANUAL CODE: CPI: A12-L04A; J01-C03; J01-D01A; J01-H  
 EPI: S03-E09C; S03-E09D

L40 ANSWER 7 OF 21 WPIX COPYRIGHT 2009 THOMSON REUTERS on STN  
 ACCESSION NUMBER: 2002-627453 [67] WPIX  
 DOC. NO. CPI: C2002-177025 [67]  
 DOC. NO. NON-CPI: N2002-496141 [67]  
 TITLE: Hybrid particle for chromatographic separations, comprises interior area of specified composition and exterior surface comprising another composition plus the previous composition  
 DERWENT CLASS: A26; A97; E11; J01; P73  
 INVENTOR: O'GARA J E; OGARA J E  
 PATENT ASSIGNEE: (OGAR-I) O'GARA J E; (WATE-N) WATERS INVESTMENTS LTD  
 COUNTRY COUNT: 99

## PATENT INFORMATION:

PATENT NO	KIND DATE	WEEK	LA	PG	MAIN IPC
WO 2002060562	A1 20020808	(200267)*	EN	53[0]	
<--					
US 20020147293	A1 20021010	(200269)	EN		
<--					
US 6528167	B2 20030304	(200320)	EN		
EP 1361915	A1 20031119	(200377)	EN		
US 20040048067	A1 20040311	(200419)	EN		
AU 2002243723	A1 20020812	(200427)	EN		
<--					
JP 2004532975	W 20041028	(200471)	JA	93	
US 7175913	B2 20070213	(200714)	EN		
US 20080053894	A1 20080306	(200819)	EN		
JP 2009080128	A 20090416	(200926)	JA	40	
JP 4282324	B2 20090617	(200940)	JA	36	

## APPLICATION DETAILS:

PATENT NO	KIND	APPLICATION	DATE
WO 2002060562	A1	WO 2002-US2678	20020129
US 20020147293	A1	US 2001-774533	20010131
US 6528167	B2	US 2001-774533	20010131
US 20040048067	A1 Cont of	US 2001-774533	20010131
US 7175913	B2 Cont of	US 2001-774533	20010131
US 20080053894	A1 Cont of	US 2001-774533	20010131
AU 2002243723	A1	AU 2002-243723	20020129
EP 1361915	A1	EP 2002-709226	20020129
JP 2004532975	W	JP 2002-560751	20020129

JP 2009080128 A Div Ex	JP 2002-560751 20020129
EP 1361915 A1	WO 2002-US2678 20020129
JP 2004532975 W	WO 2002-US2678 20020129
US 20040048067 A1	US 2003-352582 20030128
US 7175913 B2	US 2003-352582 20030128
US 20080053894 A1 Cont of	US 2003-352582 20030128
US 20080053894 A1	US 2006-607301 20061130
JP 2009080128 A	JP 2008-295087 20081119
JP 4282324 B2	JP 2002-560751 20020129
JP 4282324 B2 PCT Application	WO 2002-US2678 20020129

## FILING DETAILS:

PATENT NO	KIND	PATENT NO
US 20040048067	A1 Cont of	US 6528167 B
US 7175913	B2 Cont of	US 6528167 B
US 20080053894	A1 Cont of	US 6528167 B
US 20080053894	A1 Cont of	US 7175913 B
EP 1361915	A1 Based on	WO 2002060562 A
AU 2002243723	A1 Based on	WO 2002060562 A
JP 2004532975	W Based on	WO 2002060562 A
JP 4282324	B2 Previous Publ	JP 2004532975 W
JP 4282324	B2 Based on	WO 2002060562 A

PRIORITY APPLN. INFO: US 2001-774533 20010131  
 US 2003-352582 20030128  
 US 2006-607301 20061130

## INT. PATENT CLASSIF.:

MAIN: B01D0053-00; G01N0030-48  
 SECONDARY: B01J0020-26; B32B0003-00; C08G0077-04;  
 C08J0003-12  
 IPC ORIGINAL: B01D0015-08 [N,A]; B01D0015-08 [N,C];  
 B01J0020-281 [I,A]; B01J0020-281  
 [I,C]; B32B0001-00 [I,A]; B32B0001-00 [I,C];  
 C08G0077-00 [I,C]; C08G0077-04 [I,A]; C08J0003-12  
 [I,A]; C08J0003-12 [I,C]; C08K0009-00 [I,C];  
 C08K0009-10 [I,A]; G01N0030-00 [I,C]; G01N0030-00  
 [I,C]; G01N0030-88 [I,A]

IPC RECLASSIF.: B01D0015-08 [I,A]; B01D0015-08 [I,C];  
 B01J0020-10 [I,A]; B01J0020-10  
 [I,C]; B01J0020-22 [I,C];  
 B01J0020-26 [I,A]; B01J0020-281  
 [I,A]; B01J0020-281 [I,C];  
 B01J0020-286 [I,A]; B01J0020-30  
 [I,C]; B01J0020-32 [I,A]; C08G0077-00 [I,C]  
 ; C08G0077-04 [I,A]; C08J0003-12 [I,A]; C08J0003-12  
 [I,C]; G01N0030-00 [I,C]; G01N0030-88 [I,A]  
 B01J0020-10B; B01J0020-26; B01J0020-32  
 210/502.100; 428/405.000; 528/010.000  
 ECLA: 055/524.000; 096/101.000; 096/108.000; 096/290.000;  
 USCLASS NCLM: 428/402.000; 428/403.000; 428/404.000; 523/206.000  
 NCLS:

## JAP. PATENT CLASSIF.:

MAIN/SEC.: B01D0015-08; B01J0020-26 L; C08G0077-04; G01N0030-48  
 G; G01N0030-48 L; G01N0030-88 101 C; G01N0030-88 101  
 L; G01N0030-88 101 P; G01N0030-88 201 G; G01N0030-88  
 201 X; C08J0003-12 Z (CFH)  
 MAIN: G01N0030-48 L; G01N0030-88 101 L; G01N0030-88 201 X  
 SECONDARY: B01J0020-26 L; C08G0077-04; G01N0030-48 G;  
 G01N0030-88 101 C; G01N0030-88 201 G; C08J0003-12 Z

(CFH)  
 B01D0015-08  
 INDEX: C08L0083:04  
 FTERM CLASSIF.:  
 2G063; 4D017; 4F070; 4G066; 4J035; 4J246; 4J246/AA02;  
 4D017/AA03; 4J246/AA03; 4F070/AA59; 4F070/AA60;  
 4G066/AB01.A; 4G066/AB03.A; 4G066/AB05.A;  
 4G066/AB06.A; 4G066/AB07.A; 4G066/AB09.A;  
 4G066/AB10.A; 4G066/AB12.A; 4G066/AB15.A; 4J246/AB15;  
 4G066/AB18.A; 4G066/AB21.A; 4G066/AC11.A;  
 4G066/AC28.A; 4G066/AC28.B; 4G066/AC35.B;  
 4G066/AD13.B; 4J246/BA01.0; 4D017/BA03; 4J246/BA05.X;  
 4J246/BA07.0; 4G066/BA09; 4J246/BA11.0; 4J246/BA11.X;  
 4J246/BA16.0; 4J246/BA16.X; 4J246/BA17.0;  
 4J246/BA17.X; 4G066/BA20; 4J246/BA21.0; 4G066/BA23;  
 4G066/BA25; 4G066/BA26; 4J246/BA36.0; 4J246/BA37.0;  
 4J246/BB02.0; 4J246/BB02.X; 4D017/CA01; 4J246/CA04.0;  
 4J246/CA04.M; 4J246/CA05.M; 4J246/CA12.0;  
 4J246/CA12.M; 4J246/CA13.0; 4J246/CA14.0;  
 4J246/CA23.0; 4J246/CA24.U; 4J246/CA25.0;  
 4J246/CA26.0; 4J246/CA35.0; 4J246/CA36.0;  
 4J246/CA39.0; 4J246/CA52.0; 4J246/CA58.0;  
 4J246/CA72.0; 4J246/CA74.0; 4J246/CA76.0;  
 4J246/CA78.0; 4J246/CA99.0; 4J246/CB01; 4J246/CB02;  
 4J246/CB03; 4D017/DA03; 4G066/DA07; 4F070/DA32;  
 4F070/DA40; 4F070/DC03; 4F070/DC15; 4G066/EA01;  
 4J246/EA28; 4J246/FA07.1; 4G066/FA09; 4G066/FA21;  
 4J246/FA42.1; 4J246/FC21.2; 4J246/FD06; 4G066/GA11;  
 4J246/GA11; 4J246/GA13; 4J246/GB18; 4J246/GB24;  
 4J246/GB32

## BASIC ABSTRACT:

WO 2002060562 A1 UPAB: 20090430

NOVELTY - A hybrid particle comprises an interior area having a specified composition and an exterior surface comprising another composition (1-99%) and a composition (balance) same as that of interior area.

DETAILED DESCRIPTION - A hybrid particle having an interior area and an exterior surface comprises a composition of formula (I). The interior area comprises composition A. The exterior surface composition comprises composition B (1-99%) and composition A (balance).

(Ay)(B)x (I)

SiO<sub>2</sub>/(R1pR2qSiOt)n (II)

SiO<sub>2</sub>/(R3(R1rSiOt)m)n (III)

SiO<sub>2</sub>/(R4vSiOt)n (IV)

x, y = whole number integers;

A = compound of formula (II) and/or (III);

R1, R2 = (substituted) 1-7C alkyl or (substituted) aryl;

R3 = (substituted) 1-7 alkylene, alkenylene, alkenylene or arylene

bridging silicon atoms;

p, q = 0-2;

p+q = 1 or 2;

r = 0 or 1;

m = at least 2;

n = 0.01-100;

B = compound of formula (IV);

R4 = OH, fluorine, alkoxy, aryloxy, substituted siloxane, protein peptide, carbohydrate and/or nucleic acid;

v = 1 or 2; and

n = 0.01-100.

Provided that when p+q is 1, t is 1.5 and when p+q is 2, t is 1. When r is 0, t is 1.5 and when r is 1, t is 1. When v is 1, t is 1.5 and when v is 2, t is 1. R4 is not R1, R2 or R3.

INDEPENDENT CLAIMS are included for:

- (a) A bulk material comprising the particles above;
- (b) A method of performing a chromatographic separation comprising running a sample through a column containing particles;
- (c) A separation device comprising the particle above;
- (d) A method of preparing hybrid chromatographic particles comprising prepolymerizing an organoalkoxysilane and/or tetraalkoxysilane in the presence of an acid catalyst to give polyalkoxysiloxane, preparing an aqueous suspension of the polyalkoxysilane comprising a surfactant and gelling in the presence of a base catalyst to give porous particles having 1-7C alkyl, (substituted) aryl, 1-7C alkylene, alkenylene, alkynylene or arylene, modifying the pore structure of the porous particles by hydrothermal treatment, and replacing one or more 1-7C alkyl, (substituted) aryl, 1-7C alkylene, alkenylene, alkynylene or arylene of the particle with hydroxyl, fluorine, alkoxy, aryloxy or substituted siloxane; and
- (e) A method of forming a porous inorganic/organic hybrid material comprising forming a porous inorganic/organic hybrid particle having surface silicon-methyl groups, replacing surface silicon-methyl groups of the hybrid particle with hydroxyl groups, bonding alkyl groups to the surface of the porous inorganic/organic hybrid particle, replacing surface silicon-methyl groups with fluorine groups, and end-capping the surface of the hybrid particle with trimethylchlorosilane.

USE - The hybrid particles are used for performing chromatographic separations, or for participating in chemical reactions.

ADVANTAGE - The inventive hybrid particle demonstrates higher bonded phase surface concentrations, improved stability and separation characteristics, improved low pH stability and improved chromatographic separation performance.

TECHNOLOGY FOCUS:

INSTRUMENTATION AND TESTING - Preferred Device: The separation device comprises chromatographic columns, filtration membranes, sample clean up devices or microtiter plates.

POLYMERS - Preferred Method: In the method of preparing hybrid chromatographic particles, the replacing step involves reacting the hybrid particle with aqueous hydrogen peroxide, potassium fluoride and potassium bicarbonate in an organic solution and modifying the surface of the hybrid chromatographic particles with a surface modifier. The method further comprises end-capping the surface of the hybrid particles.

ORGANIC CHEMISTRY - Preferred Composition: The exterior surface has a composition comprising composition B (70-90%, preferably 80-90%) and composition A (balance). Preferred Properties: The particles have a mean particle size of 0.5-100 microm, preferably 1-20 microm. It is surface modified by a polymer coating and has a surface concentration of R6 greater than 2.5 micromol/m<sup>2</sup>, preferably greater than 3.7 micromol/m<sup>2</sup>. The molar ratio of the organotrialkoxysilane and tetraalkoxysilane is 100:1-0.01:1. The particle has a specific surface area of 50-800 m<sup>2</sup>/g, preferably 100-200 m<sup>2</sup>/g. It has a specific pore volume of 0.25-1.5 cm<sup>3</sup>/g, preferably 0.5-1.0 cm<sup>3</sup>/g and an average pore diameter of 50-500 Angstrom, preferably 100-300 Angstrom.

Preferred Component: The surfactant is an alkylphenoxypolyethoxyethanol. The suspension further comprises a porogen. The surface modifier is a haloorganosilane or a compound of formula (V).

Za(R')bSi-R (V)

Z = Cl, Br, I, 1-5C alkoxy, dialkylamino;

a, b = 0-3;

a+b = 3;

R' = 1-6C straight, cyclic or branched alkyl;

R = functional group, preferably alkyl, aryl, cyano, amino,

diol, nitro, cation or anion exchange groups or embedded polar functionalities; and

R' = methyl, ethyl, propyl, isopropyl, butyl, t-butyl, sec-butyl, pentyl, isopentyl, hexyl or cyclohexyl.

## EXTENSION ABSTRACT:

DEFINITIONS - Preferred Definition: - R4 = hydroxyl, fluorine, methoxy or -OSi(R5)b-R6; - R5 = 1-6 C straight, cyclic or branched alkyl, aryl or alkoxy or silane; - R6 = 1-36C straight, cyclic or branched alkyl, aryl or alkoxy (substituted) with halogen, cyano, amino, diol, nitro, ether, carbonyl, epoxide, sulfonyl, cation exchanger, anion exchanger, carbamate, amide, urea, peptide, protein, carbohydrate and/or nucleic acid functionalities, 18C group or cyanopropyl.

SPECIFIC COMPOUNDS - Specific Silanes: The tetraalkoxysilane is tetramethoxysilane or tetraethoxysilane. The haloorganosilane is octyldimethylchlorosilane or octadecyldimethylchlorosilane.

EXAMPLE - A mixture of TRITON X-45 (RTM: non-ionic surfactant) (24 g), ethanol (285 ml) and deionized water (1200 ml) was heated at 55degreesC for 0.5h giving a white liquid. Under rapid agitation, a solution containing toluene (60 ml) in polymethylethoxysiloxane (249 g) was added into the ethanol/water TRITON X-45 mixture and emulsified in the aqueous phase. Ammonium hydroxide (190 ml) was added to the emulsion to gel the emulsion beads.

The gelled product was stirred at 55degreesC for 16h.

FILE SEGMENT: CPI; GMPI  
MANUAL CODE: CPI: A06-A00E; A12-L04A; E05-E01; E05-E02C; E11-Q03E; E31-P03; J01-D01A

L40 ANSWER 8 OF 21 WPIX COPYRIGHT 2009 THOMSON REUTERS on STN  
ACCESSION NUMBER: 2002-616997 [66] WPIX  
DOC. NO. CPI: C2002-174415 [66]  
DOC. NO. NON-CPI: N2002-488215 [66]  
TITLE: Fractionating RNA according to size, useful for preparing cDNA libraries containing large inserts, by elution from a non-polar separation medium  
DERWENT CLASS: B04; D16; S03  
INVENTOR: AZARANI A; GJERDE D T; HANNA C P; HECKER K H; HORNBY D; MATIN M  
PATENT ASSIGNEE: (AZAR-I) AZARANI A; (GJER-I) GJERDE D T; (HANN-I) HANNA C P; (HECK-I) HECKER K H; (HORN-I) HORNBY D; (MATI-I) MATIN M; (TRAN-N) TRANSGENOMIC INC  
COUNTRY COUNT: 1

## PATENT INFORMATION:

PATENT NO	KIND	DATE	WEEK	LA	PG	MAIN IPC
US 20020062017	A1	20020523	(200266)*	EN	30[8]	
US 6521411	B2	20030218	(200317)	EN		

## APPLICATION DETAILS:

PATENT NO	KIND	APPLICATION	DATE
US 20020062017	A1	Provisional	US 2000-236398P 20000928
US 20020062017	A1	Provisional	US 2000-250306P 20001129
US 20020062017	A1	Provisional	US 2000-256050P 20001215
US 20020062017	A1		US 2001-808447 20010313

PRIORITY APPLN. INFO: US 2001-898447 20010313  
US 2000-236398F 20000928  
US 2000-250306F 20001129  
US 2000-256050F 20001215

## INT. PATENT CLASSIF.:

IPC RECLASSIF.: C12N0015-10 [I,A]; C12N0015-10 [I,C]

ECLA: C12N0015-10D

USCLASS NCLM: 536/025.400

NCLS: 435/006.000; 435/007.100; 435/320.100

## BASIC ABSTRACT:

US 20020062017 A1 UPAB: 20050526

NOVELTY - Preparing a fraction (A) of mRNA molecules (I) for producing a cDNA library that is enriched in inserts of a selected relative size range.

DETAILED DESCRIPTION - (I) of various sizes are applied to a separation medium, having a non-polar surface, in presence of a counterion agent, and at least some (I) are eluted using a mobile phase that includes a solvent less polar than water, so as to fractionate (I) at least partly according to size. Fractions of the eluate, enriched for (I) of a particular size range are then collected.

An INDEPENDENT CLAIM is also included for a cDNA library enriched for inserts of a selected relative size range.

USE - (A) is especially used to prepare libraries of high molecular weight cDNA.

ADVANTAGE - Enrichment for large mRNA eliminates cloning competition from small fragments, making it more likely that large mRNA (corresponding to complete genes) will be cloned. Also fractionated RNA is more stable (possibly associated with lack of ribonucleases), and rare, low copy number, molecules are efficiently recovered.

## TECHNOLOGY FOCUS:

BIOTECHNOLOGY - Preferred Process: The fraction of mRNA collected is enriched for larger molecules, and the starting material is total (m)RNA from a biological sample. Separation is particularly by ion-pairing, reverse phase high performance liquid chromatography, in a batch process that uses a spin column, vacuum tray device, or low/medium pressure column, optionally under denaturing conditions provided by:

- (i) temperature above 50 (preferably 75)degreesC;
- (ii) chemical denaturant; or
- (iii) denaturing pH.

Particularly separation is performed in absence of multivalent cations that might interfere with nucleic acid separation.

Preferred Materials: The separation medium comprises particles of an (in)organic material or insoluble polysaccharide coated with a hydrocarbon or non-polar hydrocarbon-substituted polymer in which any polar groups are reacted with a non-polar (substituted) hydrocarbon, particularly polymeric beads of average diameter 0.5-100, best 1-3, micron. Alternatively, the medium is a monolith.

Preferred Process: Elution is with a reagent free of multivalent cations, or the medium is subjected to an acid wash to remove such cations or treated with an agent that binds them.

Preferred Library: This comprises cDNA inserts, produced by reverse transcription from the selected RNA fractions, in vectors, particularly phages or plasmids, and are especially present in a host cell.

INORGANIC CHEMISTRY - Preferred Materials: Typical of many suitable support materials are silica, silica carbide, silica nitrate, titanium oxide, aluminium oxide, zirconium oxide, alumina and carbon.

POLYMERS - Preferred Materials: The separation beads are particularly made of C18-alkylated, non-porous

poly(styrene-divinylbenzene).

ORGANIC CHEMISTRY - Preferred Materials: The mobile phase for fractionation contains an organic solvent, specifically acetonitrile and a counterion agent. The counterion agent is selected from octylammonium acetate, octadimethylammonium acetate, decylammonium acetate, actadecylammonium acetate, pyridiniumammonium acetate, cyclohexylammonium acetate, diethylammonium acetate, propylethylammonium acetate, propyldiethylammonium acetate, butylethylammonium acetate, methylhexylammonium acetate, tetramethylammonium acetate, tetrapropylammonium acetate, tetrabutylammonium acetate, dimethyldiethylammonium acetate, triethylammonium acetate, tripropylammonium acetate, tributylammonium acetate, tetrapropylammonium acetate, tetrabutylammonium acetate, triethylammonium hexafluoroisopropyl alcohol and or mixtures of the above, most preferably tetrabutylammonium bromide or triethylammonium acetate. Particularly the mobile phase has pH 5-9, best 6-7.5.

EXTENSION ABSTRACT:

EXAMPLE - A sample (5 mug) of human fetal liver mRNA was separated by ion-pairing, reverse phase high performance liquid chromatography, using a commercial column (7.8 mm diameter; 50 mm long) containing non-polar C18-alkylated poly(styrene-divinylbenzene). Fractionation was performed at 75degreesC (denaturing conditions) using 0.9 ml/min of a step gradient of eluents (A), 0.1 M triethylammonium acetate, pH 7 and (B) 0.1 M TEM (not defined) in 25% acetonitrile in water, and the eluate monitored at 260 nm. After 6 min, fractions were taken every minute, reverse transcribed, the cDNA attached to EcoRI linkers, amplified and the amplicons analyzed by gel electrophoresis. For fractions taken after 10, 11 and 12 min, respectively, the cDNA consisted mainly of sequences of about 500 bp; 1 kb and 3-10 kb, and fragments larger than 10 kb were recovered after 13-15 min. When non-fractionated mRNA was tested similarly, the cDNA was mostly 1.5-5 kb.

FILE SEGMENT: CPI; EPI  
MANUAL CODE: CPI: B04-C02; B04-E02; B04-E03; B04-E05; B04-E08;  
B04-F01; B04-F0100E; B05-A03B; B05-E02C;  
B05-C03; B05-C06; B05-C08; B07-D04A; B10-A22;  
B11-B; B11-C09; D05-H12; D05-H12D; D05-H12E;  
D05-H14  
EPI: S03-E09C5; S03-E14H

L40 ANSWER 9 OF 21 WPIX COPYRIGHT 2009 THOMSON REUTERS on STN  
ACCESSION NUMBER: 2001-050851 [07] WPIX  
DOC. NO. CPI: C2001-014217 [07]  
TITLE: Continuous production of porous or non-  
porous bead polymer, e.g.  
polystyrene, acrylic or silica gel  
, involves dispersing component streams in a static  
micro-mixer and reacting  
DERWENT CLASS: A18; A89; B04; J04  
INVENTOR: EISENBEISS F; KINKEL J; MUELLER H  
PATENT ASSIGNEE: (MERE-C) MERCK PATENT GMBH  
COUNTRY COUNT: 90

PATENT INFORMATION:

PATENT NO	KIND DATE	WEEK	LA	PG	MAIN IPC
DE 19920794	A1	20001109	(200107)*	DE	13[6]

<--



December 17, 2009

10/536,853

33

WO 2000068300	A1	20001116	(200107)	DE
<---				
AU 2000049135	A	20001121	(200112)	EN
<---				
EP 1177243	A1	20020206	(200218)	DE
<---				
US 6492471	B1	20021210	(200301)	EN
<---				
JP 2002544309	W	20021224	(200313)	JA 29
<---				
EP 1177243	B1	20030402	(200325)	DE
DE 50001620	G	20030508	(200332)	DE

## APPLICATION DETAILS:

PATENT NO	KIND	APPLICATION	DATE
DE 19920794 A1		DE 1999-19920794	
19990506			
AU 2000049135 A		AU 2000-49135	20000425
DE 50001620 G		DE 2000-50001620	
20000425			
EP 1177243 A1		EP 2000-931070	20000425
EP 1177243 B1		EP 2000-931070	20000425
DE 50001620 G		EP 2000-931070	20000425
JP 2002544309 W		JP 2000-616269	20000425
WO 2000068300 A1		WO 2000-EP3677	20000425
EP 1177243 A1		WO 2000-EP3677	20000425
US 6492471 B1		WO 2000-EP3677	20000425
JP 2002544309 W		WO 2000-EP3677	20000425
EP 1177243 B1		WO 2000-EP3677	20000425
DE 50001620 G		WO 2000-EP3677	20000425
US 6492471 B1		US 2001-959679	20011105

## FILING DETAILS:

PATENT NO	KIND	PATENT NO
DE 50001620 G	Based on	EP 1177243 A
AU 2000049135 A	Based on	WO 2000068300 A
EP 1177243 A1	Based on	WO 2000068300 A
US 6492471 B1	Based on	WO 2000068300 A
JP 2002544309 W	Based on	WO 2000068300 A
EP 1177243 B1	Based on	WO 2000068300 A
DE 50001620 G	Based on	WO 2000068300 A

PRIORITY APPLN. INFO: DE 1999-19920794 19990506  
 INT. PATENT CLASSIF.:

MAIN: C08F0002-01; C08J0003-14  
 B01J0019-00; C01B0033-152  
 SECONDARY: B01J0020-30 [I,A]; B01J0020-30  
 IPC RECLASSIF.: [I,C]; C01B0033-00 [I,C]; C01B0033-14 [I,A];  
 C08F0002-00 [I,A]; C08F0002-00 [I,C]; C08F0002-01  
 [I,A]; C08F0002-01 [I,C]; C08F0002-04 [I,C];  
 C08F0002-10 [I,A]; C08F0002-12 [I,A]; C08F0002-12  
 [I,C]; C08F0002-22 [I,A]; C08G0085-00 [I,A];  
 C08G0085-00 [I,C]  
 ECLA: B01J0019-00R; C08F0002-01; C08F0002-10  
 ICO: L01F0013:00M2C; L01J0219:00C2L2; L01J0219:00R2B;  
 L01J0219:00R2H2; L01J0219:00R4B

USCLASS NCLM: 526/088.000  
NCLS: 526/317.100; 526/329.700; 526/336.000; 526/909.000;  
528/010.000; 528/425.000; 536/063.000

JAP. PATENT CLASSIF.:  
MAIN/SEC.: B01J0020-30; C01B0033-14; C08F0002-01; C08G0085-00  
FTERM CLASSIF.: 4G066; 4G072; 4J011; 4J031; 4J011/AA08; 4G066/AA22.B;  
4G072/AA28; 4G066/AC02.B; 4J011/AC06; 4G066/AC14.B;  
4G066/BA09; 4G066/BA20; 4G072/BB05; 4G072/BB07;  
4G072/BB15; 4J031/CA07; 4J031/CA22; 4J031/CA27;  
4J031/CA39; 4J031/CA42; 4G072/CC10; 4J031/CE10;  
4J031/CG03; 4G066/EA01; 4G066/FA07; 4G066/FA08;  
4G072/HH30; 4J011/JB08; 4J011/JB09; 4J011/JB11;  
4J011/JB14; 4J011/JB26; 4J011/KA27; 4J011/KB08;  
4J011/KB11; 4J011/KB14; 4J011/KB29; 4G072/MM01;  
4G072/MM02; 4G072/RR12; 4G072/UU11; 4G072/UU17

## BASIC ABSTRACT:

DE 19920794 A1 UPAB: 20050524

NOVELTY - A process for the production of porous or non-porous bead polymers involves dispersing at least two liquid streams of component solutions in a static micro-mixer and then reacting the components.

DETAILED DESCRIPTION - An INDEPENDENT CLAIM is included for powder consisting of porous or non-porous spherical bead polymers produced by this process, with a narrow particle size range between 0.1 and 300 microns.

USE - For the production of bead polymers by radical polymerisation or polycondensation, e.g. acrylic polymers, styrene polymers, polyesters, polyamides, polyurethanes, silica gel. Bead polymers are used, e.g. as ion exchange or adsorber resins, chromatographic supports or catalyst supports, polymeric reagents, imprinting polymers and support materials for combinatorial chemistry and synthesis of peptides or oligonucleotides.

ADVANTAGE - The use of new high-performance micro-mixers in combination with a simple reactor enables the continuous production of higher yields of bead polymers with a narrow, controllable particle size range and more uniform quality. Typically, this process gives yields of almost 100% in the required size range, compared with 30% for polymerizations carried out in a stirred reactor.

DESCRIPTION OF DRAWINGS - Diagram of high-performance micro-mixer.

base plate; (1)

cover plate; (2)

layer of 15 or 18 sinusoidal channels with a width of 25 or 40 microns;

(3)

component feed streams; (4a, 4b)

outlet slot on top of mixer unit (5)

## TECHNOLOGY FOCUS:

POLYMERS - Preferred Process: One of more accessory devices may be connected before and/or after the micro-mixer. The component solutions contain no emulsifiers or protective colloids.

Preferred Mixer: The LIGA Micromixing System (RTM: micro-mixer) is preferred.

## EXTENSION ABSTRACT:

EXAMPLE - A high-performance micro-mixer (IMM Type A7; channel width 40 microns) was filled with water and then supplied with: - (A) a 1:1 mixture of styrene and divinylbenzene containing 0.2 wt% azobis-(isobutyronitrile); and - (B) water; - from two separate storage containers (both at 40 degrees C) via two pumps, with constant flow rates in a ratio (A:B) of 1:10 by volume. The mixture obtained was then passed downwards through a vertical tubular reactor with 3 sections (each with a length of 10 m and with internal diameters of 2, 5 and 10 mm respectively) which was heated to 85 degrees C and finally into a stirred reactor with a

slow-speed stirrer. The product was a heterogeneously crosslinked styrene-divinylbenzene bead polymer with a narrow size distribution.

FILE SEGMENT: CPI  
MANUAL CODE: CPI: A10-B05; B04-C03D; B12-M11D; J04-B01C;  
J04-E03

L40 ANSWER 10 OF 21 WPIX COPYRIGHT 2009 THOMSON REUTERS on STN  
ACCESSION NUMBER: 2000-558126 [51] WPIX  
CROSS REFERENCE: 2002-673309; 2004-708502; 2008-C16185; 2009-J62451  
TITLE: New porous inorganic/organic hybrid  
particle having chromatographically  
-enhancing pore geometry for chromatographic  
separations  
DERWENT CLASS: A26; A89; J04; P42; P73  
INVENTOR: FISK R; FISK R P; JIANG Z; WALTER T; WALTER T H;  
ZHIPING J; FISK P; WALTER H  
PATENT ASSIGNEE: (WATE-N) WATERS INVESTMENTS LTD  
COUNTRY COUNT: 84

## PATENT INFORMATION:

PATENT NO	KIND	DATE	WEEK	LA	PG	MAIN IPC
WO 2000045951	A1	20000810	(200051)*	EN	37[0]	
<--						
AU 2000028721	A	20000825	(200059)	EN		
<--						
US 20010033931	A1	20011025	(200170)	EN		
<--						
EP 1163050	A1	20011219	(200206)	EN		
<--						
JP 2002536630	W	20021029	(200274)	JA	38	
<--						
EP 1733786	A1	20061220	(200702)	EN		
EP 1163050	B1	20070103	(200703)	EN		
DE 60032750	E	20070215	(200724)	DE		
DE 60032750	T2	20071108	(200774)	DE		

## APPLICATION DETAILS:

PATENT NO	KIND	APPLICATION	DATE
WO 2000045951 A1		WO 2000-US3052	20000204
US 20010033931 A1 Cont of		US 1999-244795	19990205
AU 2000028721 A		AU 2000-28721	20000204
DE 60032750 E		DE 2000-632750	20000204
EP 1163050 A1		EP 2000-907186	20000204
EP 1733786 A1 Div Ex		EP 2000-907186	20000204
EP 1163050 B1		EP 2000-907186	20000204
DE 60032750 E		EP 2000-907186	20000204
JP 2002536630 W		JP 2000-597060	20000204
EP 1163050 A1		WO 2000-US3052	20000204
JP 2002536630 W		WO 2000-US3052	20000204
EP 1163050 B1		WO 2000-US3052	20000204
DE 60032750 E		WO 2000-US3052	20000204
US 20010033931 A1		US 2001-858087	20010514
EP 1733786 A1		EP 2006-14856	20000204
EP 1163050 B1 Related to		EP 2006-14856	20060717
DE 60032750 T2		DE 2000-632750	20000204

DE 60032750 T2  
DE 60032750 T2

EP 2000-907186 20000204  
WO 2000-US3052 20000204

## FILING DETAILS:

PATENT NO	KIND		PATENT NO	
EP 1733786	A1	Div ex	EP 1163050	A
DE 60032750	E	Based on	EP 1163050	A
EP 1163050	B1	Related to	EP 1733786	A
AU 2000028721	A	Based on	WO 2000045951	A
EP 1163050	A1	Based on	WO 2000045951	A
JP 2002536630	W	Based on	WO 2000045951	A
EP 1163050	B1	Based on	WO 2000045951	A
DE 60032750	E	Based on	WO 2000045951	A
DE 60032750	T2	Based on	EP 1163050	A
DE 60032750	T2	Based on	WO 2000045951	A

PRIORITY APPLN. INFO: US 1999-244795 19990205  
US 2001-858087 20010514

## INT. PATENT CLASSIF.:

MAIN: G01N0030-48  
SECONDARY: B01J0020-22  
IPC ORIGINAL: B01J0020-10 [I,A]; B01J0020-10  
[I,C]; B01J0020-10 [I,A];  
B01J0020-10 [I,C]; B01J0020-22  
[I,A]; B01J0020-22 [I,C];  
B01J0020-22 [I,A]; B01J0020-22  
[I,C]; B01J0021-00 [I,C]; B01J0021-00 [I,C];  
B01J0021-00 [I,C]; B01J0021-06 [I,A]; B01J0021-06  
[I,A]; B01J0021-08 [I,A]; B01J0021-08 [I,A];  
B01J0021-12 [I,A]; B01J0021-12 [I,A]; B01J0031-02  
[I,A]; B01J0031-02 [I,C]; B01J0031-02 [I,A];  
B01J0031-02 [I,C]; B01J0031-06 [I,A]; B01J0031-06  
[I,C]; B01J0031-06 [I,A]; B01J0031-06 [I,C];  
B05D0007-00 [I,A]; B05D0007-00 [I,C]; B05D0007-00  
[I,A]; B05D0007-00 [I,C]; B32B0005-16 [I,A];  
B32B0005-16 [I,C]; B32B0005-16 [I,A]; B32B0005-16  
[I,C]; C07F0005-00 [I,C]; C07F0005-00 [I,C];  
C07F0005-06 [I,A]; C07F0005-06 [I,A]; C07F0007-00  
[I,C]; C07F0007-00 [I,C]; C07F0007-02 [I,A];  
C07F0007-02 [I,A]; C08F0004-00 [I,A]; C08F0004-00  
[I,C]; C08F0004-00 [I,A]; C08F0004-00 [I,C]

## IPC RECLASSIF.:

B01J0020-10 [I,A]; B01J0020-10  
[I,C]; B01J0020-281 [I,A];  
B01J0020-281 [I,C]; B01J0020-283  
[I,A]; B01J0020-284 [I,A];  
B01J0020-286 [I,A]; B01J0020-289  
[I,A]; B01J0020-30 [I,C];  
B01J0020-32 [I,A]; B01J0039-26 [I,A];  
B01J0039-26 [I,C]; B01J0041-20 [I,A]; B01J0041-20  
[I,C]; C08F0004-00 [I,A]; C08F0004-00 [I,C];  
G01N0030-00 [I,C]; G01N0030-88 [I,A]  
B01J0020-10B; B01J0020-286; B01J0020-289;  
B01J0020-32; B01J0039-26; B01J0041-20

## ECLA:

## ICO:

USCLASS NCLM:  
NCLS:

L01J0020:32J  
428/402.000  
427/215.000; 428/405.000

## JAP. PATENT CLASSIF.:

MAIN/SEC.: B01J0020-22 D; G01N0030-48 C; G01N0030-48 D;

G01N0030-48 E; G01N0030-48 G; G01N0030-48 H;  
 G01N0030-48 K; G01N0030-88 101 C; G01N0030-88 101 D;  
 G01N0030-88 101 E; G01N0030-88 101 H; G01N0030-88 101  
 K; G01N0030-88 101 L; G01N0030-88 201 G; G01N0030-88  
 201 X

## FTERM CLASSIF.:

2G063; 4G066; 4G066/AA20.A; 4G066/AA20.B;  
 4G066/AA22.A; 4G066/AA22.B; 4G066/AA38.A;  
 4G066/AA38.B; 4G066/AB05.A; 4G066/AB05.B;  
 4G066/AB09.A; 4G066/AB09.B; 4G066/AD06.A;  
 4G066/AD06.B; 4G066/AD10.A; 4G066/AD10.B; 4G066/BA09;  
 4G066/BA11; 4G066/BA20; 4G066/BA25; 4G066/BA26;  
 4G066/DA07; 4G066/EA01

## BASIC ABSTRACT:

WO 2000045951 A1 UPAB: 20090602

NOVELTY - A porous inorganic/organic hybrid material (A) comprises porous inorganic/organic hybrid particles having a chromatographically enhancing pore geometry.

DETAILED DESCRIPTION - INDEPENDENT CLAIMS are also included for the following:

- (a) preparation of the porous inorganic/organic hybrid particles (I) having the chromatographically enhancing pore geometry comprises:
  - (i) forming porous inorganic/organic hybrid particle; and
  - (ii) modifying the pore structure of the porous particle;
- (b) a separation device having a stationary phase containing (I); and
- (c) a chromatographic column having improved lifetime comprises:
  - (i) a column having a cylindrical interior for accepting a packing material; and
  - (ii) a packed chromatographic bed comprising porous particle of hybrid silica of formula (II) or (III). A porous particle of hybrid silica are surface modified and have an average pore diameter of 100 - 300 Angstrom .

$\text{SiO}_2/\text{R}_2\text{Pr}_4\text{qSiOt})_n$  (II)

$\text{SiO}_2/(\text{R}_6(\text{R}_2\text{rSiOt})_m)_n$  (III)

R<sub>2</sub>, R<sub>4</sub> = 1-18C alkyl or aryl moiety (optionally substituted with alkyl, cyano, amino, diol, nitro and ion exchange or embedded polar functionalities);  
 R<sub>6</sub> = 1-18C (optionally substituted) alkylene, alkenylene, alkynylene, or arylene moiety bridging two or more silicon atoms;

p and q = 0 - 2;

r = 0 - 1;

m = at least 2;

n = 0.1 - 1 (preferably 0.20 - 0.50).

When p+q = 1, t = 1.5; when p+q = 2, t = 1. when r = 0, t = 1.5 and when r = 1, t = 1.

USE - In devices such as chromatographic column, cartridges, filtration membranes, sample cleanup devices, and microtiter plates (claimed); also in thin layer chromatographic plates.

ADVANTAGE - It offers more efficient chromatographic separations than the prior art. It is chemically stable, does not have undesirable pore geometries, and shows excellent separation performance.

## TECHNOLOGY FOCUS:

POLYMERS - Preparation: (I) is prepared by prepolymerization by hydrolyzing and condensing a mixture of organoalkoxysilane (O) and a tetraalkoxysilane (T) in presence of acid catalyst to produce polyorganoalkoxysiloxane (P); preparing an aqueous suspension of (P); gelling in the presence of base catalyst (b) to produce porous particle; and modifying the pore structure of porous particle by hydrothermal treatment.

ORGANIC CHEMISTRY - Preferred Method: (I) is surface modified by polymer coating with a surface modifier (S). Any free silanol groups remaining from surface modification procedure are end capped.

Preferred Component: (S) is of the formula  $\text{Za}(\text{R}')\text{bSi-R}$ .

Z = Cl, Br, I, 1-5C alkoxy, dialkylamino or trifluoromethanesulfonate;

a and b = 0 - 3;

R' = 1-6C alkyl (preferably methyl, ethyl, propyl, isopropyl, butyl, tert-butyl, sec-butyl, pentyl, isopentyl, hexyl or cyclohexyl);

R = functionalizing group (preferably alkyl, aryl, cyano, amino, diol, nitro, a cation or anion exchange group, or an embedded polar functionality, specially 1-20C alkyl);

a + b = 3.

(S) is octyldimethylchlorosilane or octadecyldimethylchlorosilane.

(T) (preferably tetramethoxysilane or tetraethoxysilane) has the formula Si(OR<sub>5</sub>)<sub>4</sub>. (O) is of the formula:

R'<sup>3</sup>Si(OR<sub>1</sub>)<sub>3</sub> or R<sub>6</sub>(Si(OR<sub>1</sub>)<sub>3</sub>)<sub>m</sub>

R'<sup>3</sup> = 1-18C aliphatic or aromatic moiety (preferably methyl, phenyl or ethyl);

R<sub>1</sub> = 1-4C alkyl moiety (preferably ethyl);

R<sub>6</sub> = 1-18C alkylene, alkenylene, alkynylene, or arylene moiety bridging two or more silicon atoms (preferably bridging ethylene group);

m = at least 2 (preferably 2);

R<sub>5</sub> = 1-3C alkyl.

Molar ratio of (O) and (T) is 0.5:1 - 0.2:1. The aqueous suspension further comprises a porogen (preferably toluene) and surfactant (alkylphenoxypolyethoxyethanol).

INORGANIC CHEMISTRY - Preferred Particle: (I) have a mean particle size of 0.5 - 100 μm, specific surface area 50 - 800 m<sup>2</sup>/g, specific pore volume 0.25 - 1.5 cm<sup>3</sup>/g and an average pore diameter 50 - 500 Angstrom.

Preferred Components: Inorganic portion of (A) is alumina, silica, titanium or zirconium oxides or ceramic material (preferably silica). Base catalyst is free of alkali or alkaline earth metal cations.

#### EXTENSION ABSTRACT:

EXAMPLE - A mixture of Triton X-45 (RTM: surfactant) (11.8 g), Triton X-100 (RTM: surfactant) (7.8 g), ethanol (232 ml) and deionized water (980 ml) was heated at 50°C leading to white liquid. A solution containing toluene (24 ml) in poly(ethylene-bridged)ethoxysiloxane (203 g) was added to ethanol/water/triton mixture and emulsified. 30% NH<sub>4</sub>OH (154 ml) was added into the emulsion to gel the beads. The gelled product was stirred at 55°C for 16 hours, then filtered and washed with water and methanol and dried at 100°C under reduced pressure. The surface of the particles of hybrid silica produced are modified with octadecyldimethylchlorosilane (ODS) and trimethylchlorosilane. ODS (4.5 g) and imidazole (1.32 g) were added to a hybrid silica (8 g) in toluene (90 ml) and the resultant mixture was refluxed for 2 hours. The modified hybrid silica particles were filtered, washed and dried. Trimethylchlorosilane (1.65 g) and imidazole (1.32 g) were added to a mixture of ODS-modified hybrid silica in toluene (65 ml). The specific surface area, specific pore volume, ODS surface coverage and average pore diameter for the above hybrid silica was 225, 0.90, 2.15 and 123 respectively. The above derivatized hybrid silica was used for separation of mixture of neutral, polar and basic compounds. The relative retention (r) of propranolol/acenaphthene and naphthalene/acenaphthene was found to be 0.139 and 0.437 respectively. The hydrolytic stability of the columns packed by the hybrid silica were

evaluated by placing the column in 50degreesC. Water bath and flushed with 50 mM triethylamine buffer (pH = 10) in water acenaphthene was injected at flow rate of 1 ml/min. The result showed that the lifetime of the column was found to be 50 hours while the commercial column A had the lifetime of 8 hours.

FILE SEGMENT: CPI; GMP  
 MANUAL CODE: CPI: A06-A00E; A12-L04A; A12-W11A; J04-B01C

L40 ANSWER 11 OF 21 WPIX COPYRIGHT 2009 THOMSON REUTERS ON STN  
 ACCESSION NUMBER: 1999-313210 [26] WPIX  
 CROSS REFERENCE: 1998-386901; 1998-506688; 1999-009395; 1999-045150;  
 1999-070314; 1999-105524; 1999-167458; 1999-190174;  
 1999-277659; 1999-287986; 1999-457857; 2000-349558;  
 2000-375583; 2000-557659; 2000-678760; 2001-071577;  
 2001-244716; 2001-381916; 2001-463931; 2001-548990;  
 2001-611309; 2001-637908; 2001-648288; 2002-055352;  
 2002-065549; 2002-082150; 2002-204872; 2002-314786;  
 2002-424420; 2002-424729; 2002-566535; 2002-689463;  
 2002-690602; 2003-017190; 2003-093162; 2003-110192;  
 2003-196844; 2003-218954; 2003-310425; 2003-615241;  
 2003-706911; 2003-743830; 2003-777165; 2004-040958;  
 2004-051045; 2004-106502; 2004-118797; 2004-118818  
 DOC. NO. CPI: C1999-092515 [26]  
 TITLE: Separating mixture of polynucleotides useful for  
 detecting wild type sequences from mutated sequences  
 DERWENT CLASS: B04; D16; J01  
 INVENTOR: FELE R M H; GJERDE D T; HAEFELE R M; HUANG B; TANG D  
 Y; TAYLOR P D  
 PATENT ASSIGNEE: (GJER-I) GJERDE D T; (HAEF-I) HAEFELE R M; (OCCI-C)  
 OCCIDENTAL CHEM CORP; (TAYL-I) TAYLOR P D; (TRAN-N)  
 TRANSGENOMIC INC  
 COUNTRY COUNT: 81

## PATENT INFORMATION:

PATENT NO	KIND	DATE	WEEK	LA	PG	MAIN IPC
WO 9922839	A1	19990514	(199926)*	EN	58[23]	
<--						
AU 9912942	A	19990524	(199940)	EN		
<--						
US 6153810	A	20001128	(200063)	EN		
<--						
EP 1056528	A1	20001206	(200064)	EN		
<--						
JP 2001521729	W	20011113	(200204)	JA	54	
<--						
US 6342161	B1	20020129	(200210)	EN		
<--						
AU 751737	B	20020829	(200264)	EN		
<--						
US 20020158017	A1	20021031	(200274)	EN		
<--						

## APPLICATION DETAILS:

PATENT NO	KIND	APPLICATION	DATE
WO 9922839 A1		WO 1998-US23159	19981030
US 6342161 B1	Provisional	US 1997-64428P	19971030

US 20020158017 A1 Provisional	US 1997-64428P 19971030
US 6342161 B1 Provisional	US 1998-70467P 19980105
US 20020158017 A1 Provisional	US 1998-70467P 19980105
US 6342161 B1 CIP of	US 1998-58337 19980410
US 6342161 B1 CIP of	US 1998-58580 19980410
US 6153810 A	US 1998-70467 19980430
US 6342161 B1 CIP of	US 1998-129105 19980804
US 6342161 B1 Provisional	US 1998-103313P 19981006
US 20020158017 A1 Provisional	US 1998-103313P 19981006
EP 1056528 A1	EP 1998-956414 19981030
US 6342161 B1 Div Ex	US 1998-183047 19981030
EP 1056528 A1	WO 1998-US23159 19981030
JP 2001521729 W	WO 1998-US23159 19981030
AU 9912942 A	AU 1999-12942 19981030
AU 751737 B	AU 1999-12942 19981030
JP 2001521729 W	JP 2000-518763 19981030
US 6342161 B1	US 2000-481476 20000111
US 20020158017 A1 Cont of	US 2000-481476 20000111
US 20020158017 A1	US 2002-54214 20020121

## FILING DETAILS:

PATENT NO	KIND	PATENT NO
AU 751737 B	Previous Publ	AU 9912942 A
US 6342161 B1	Div ex	US 6024878 A
US 6342161 B1	CIP of	US 6287822 A
US 20020158017 A1	Cont of	US 6342161 B
AU 9912942 A	Based on	WO 9922839 A
EP 1056528 A1	Based on	WO 9922839 A
JP 2001521729 W	Based on	WO 9922839 A
AU 751737 B	Based on	WO 9922839 A

PRIORITY APPLN. INFO: US 1998-103313P 19981006

US 1997-64428P	19971030
US 1998-70467P	19980105
US 1998-58337	19980410
US 1998-58580	19980410
US 1998-129105	19980804
US 1998-70467	19980430
US 1998-183047	19981030
US 2000-481476	20000111
US 2002-54214	20020121

## INT. PATENT CLASSIF.:

MAIN: C12N0015-09

IPC RECLASSIF.: B01D0015-08 [I,A]; B01D0015-08 [I,A]; B01D0015-08 [I,C]; B01D0015-08 [I,C]; B01J0020-22 [I,C]; B01J0020-26 [I,A]; B01J0020-30 [I,C]; B01J0020-32 [I,A]; C07C0253-00 [I,C]; C07C0253-30 [I,A]; C07C0255-00 [N,C]; C07C0255-49 [N,A]; C07C0255-51 [N,A]; C12N0015-09 [I,A]; C12N0015-09 [I,C]; C12N0015-10 [I,A]; C12N0015-10 [I,C]; C12Q0001-68 [I,A]; C12Q0001-68 [I,C]; G01N0030-00 [I,C]; G01N0030-34 [I,A]; G01N0030-88 [I,A]

ECLA: C07C0253-30

ICO: M07C0255:49; M07C0255:51

USCLASS NCLM: 210/635.000

NCLS: 210/656.000; 435/006.000; 536/025.400; 558/357.000

JAP. PATENT CLASSIF.:



MAIN/SEC.: B01D0015-08; C12N0015-00 A; G01N0030-34 E;  
G01N0030-88 101 E; G01N0030-88 101 J; G01N0030-88 101 K;  
G01N0030-88 101 N; G01N0030-88 201 G; G01N0030-88 E  
FTERM CLASSIF.: 2G063; 4B024; 4D017; 4D017/AA09; 4B024/AA11;  
4D017/AA11; 4D017/BA07; 4B024/CA04; 4D017/CA13;  
4D017/CA17; 4D017/DA03; 4D017/EA01; 4B024/HA20

## BASIC ABSTRACT:

WO 1999022839 A1 UPAB: 20050521

NOVELTY - A new method of separating a mixture of polynucleotides (PN) comprises:

(i) flowing a mixture of PN having a target range of base pairs through a column containing a separation medium having a non-polar separation surface; and

(ii) separating the mixture by eluting the column using a mobile phase having a composition which remains constant for the duration of the chromatographic separation.

DETAILED DESCRIPTION - An INDEPENDENT CLAIM is also included for a chromatographic method for separating a mixture of heteroduplex and homoduplex DNA molecules under conditions which selectively denatures a mutation site present in the heteroduplex comprising:

(i) applying the mixture to a separation column containing a medium having a non-polar separation surface and

(ii) separating the mixture by eluting the column using a mobile phase having a composition which remains constant for the duration of the chromatographic separation.

USE - The method is useful for separating polynucleotides, especially double stranded DNA fragments e.g. for detecting wild type sequences from mutated varieties which can have the same number of base pairs.

ADVANTAGE - The process can be used to separate sequences of the same length unlike gel electrophoresis and capillary gel electrophoresis which separate and analyze DNA fragments having different lengths.

## TECHNOLOGY FOCUS:

BIOTECHNOLOGY - Preferred Method: The target range of PN is 1-5000 (preferably 1-99, 100-199, 200-299, 300-399 or 400-499) base pairs. The medium comprises nonporous or porous beads of average diameter 0.5-100 microns. The beads are polymeric or are particles coated with a hydrocarbon or nonhydrocarbon substituted polymer or with all polar groups derivatized with a nonpolar hydrocarbon or substituted hydrocarbon. The surface is treated with acid wash treatment to remove multivalent cation contaminants. The mixture comprises the product of a PCR amplification or at least a portion of a preliminary mixture of polynucleotides having been separated during a preliminary elution by flowing through a column containing a separation medium having a non-polar separation surface and using preliminary elution conditions which vary during the elution (preferably a mobile phase, which varies in composition, comprises an increasing gradient of concentration of organic solvent and a counterion agent or a chelating agent).

INORGANIC CHEMISTRY - Preferred Particles: The particles comprise silica, silica carbide, silica nitride, titanium oxide, aluminum oxide, zirconium oxide, carbon, insoluble polysaccharide or diatomaceous earth.

ORGANIC CHEMISTRY - Preferred Method: The mobile phase comprises an organic solvent (preferably an alcohol, nitrile, dimethylformamide, tetrahydrofuran, ester, ether) and a counterion agent (preferably a lower alkyl primary amine, lower alkyl secondary amine, lower alkyl tertiary amine, lower alkyl trialkylammonium salt and/or quaternary ammonium salt and includes an acetate, carbonate,

phosphate, sulfate, nitrate, propionate, formate, chloride or bromide anion. 23 Specific counterions are listed e.g. octylammonium acetate.

## EXTENSION ABSTRACT:

EXAMPLE - A pUC18 DNA-HaeIII restriction enzyme digest was chromatographed on a matched ion polynucleotide chromatography column using a mobile phase comprising 14.25% acetonitrile in 0.1M triethylammonium acetate at 51 degrees Centigrade using a flow rate of 0.75 ml/min and monitored at 260 nm. The mobile phase was chosen to focus the separation on the basepair fragments in the 200-299 bp target range. The results showed an improved separation of fragments in this base pair range compared to the separation of a pUC18 DNA-HaeIII restriction enzyme digest using a gradient mobile phase.

FILE SEGMENT: CPI  
MANUAL CODE: CPI: B04-E02; D05-H09; J01-D01A

L40 ANSWER 12 OF 21 WPIX COPYRIGHT 2009 THOMSON REUTERS on STN  
ACCESSION NUMBER: 1998-347357 [30] WPIX  
CROSS REFERENCE: 2001-353575; 2002-204768  
DOC. NO. CPI: C1998-107316 [30]  
DOC. NO. NON-CPI: N1998-271132 [30]  
TITLE: A quantitative organic vapour sampler for e.g. polyaromatic hydrocarbons - uses a macromolecular resin agglomerate with a non-gel porous structure  
A89; E19; J04; S03  
DERWENT CLASS:  
INVENTOR: DAISEY J M; GUNDEL L; STEVENS R K  
PATENT ASSIGNEE: (REGC-C) UNIV CALIFORNIA  
COUNTRY COUNT: 1

## PATENT INFORMATION:

PATENT NO	KIND	DATE	WEEK	LA	PG	MAIN IPC
US 5763360	A	19980609	(199830)*	EN	26[9]	
<--						

## APPLICATION DETAILS:

PATENT NO	KIND	APPLICATION	DATE
US 5763360	A CIP of	US 1994-191344	19940202
US 5763360	A	US 1995-431358	19950428

PRIORITY APPLN. INFO: US 1995-431358 19950428  
US 1994-191344 19940202

INT. PATENT CLASSIF.:  
IPC RECLASSIF.: B01J0020-22 [I,C]; B01J0020-26 [I,A]; B01J0020-28 [I,A]; B01J0020-28 [I,C]; G01N0001-22 [N,A]; G01N0001-22 [N,C]; G01N0001-40 [I,A]; G01N0001-40 [I,C]  
ECLA: B01J0020-26; G01N0001-40  
ICO: S01N0001:22B7; S01N0001:40A

## BASIC ABSTRACT:

US 5763360 A UPAB: 20050521

A semi-volatile, organic, reversible gas sorbent used in an integrated diffusion vapour-particle sampler comprises a macroreticular resin agglomerate of randomly packed microspheres with a continuous non-gel porous structure of particles in the size range 0.05-10 microns.

USE - In integrated organic vapour-particle sampler (IOVPS) systems used to analyse e.g. laboratory atmospheres for the presence of e.g. polycyclic aromatic hydrocarbons (PAH) or for the analysis of atmospheric cigarette smoke contaminants.

ADVANTAGE - The sorbent allows efficient separation and quantitative measurement of pollutants.

#### DOCUMENTATION ABSTRACT:

US5763360

A semi-volatile, organic, reversible gas sorbent used in an integrated diffusion vapour-particle sampler comprises a macroreticular resin agglomerate of randomly packed microspheres with a continuous non-gel porous structure of particles in the size range 0.05-10 microns.

#### USE

In integrated organic vapour-particle sampler (IOVPS) systems used to analyse e.g. laboratory atmospheres for the presence of e.g. polycyclic aromatic hydrocarbons (PAH) or for the analysis of atmospheric cigarette smoke contaminants.

#### ADVANTAGE

The sorbent allows efficient separation and quantitative measurement of pollutants.

#### EXAMPLE

The apparatus was used to test for levels of cigarette smoke. The denuders used XAD-4 resin, a styrene-divinylbenzene resin with a surface area of 780 m<sup>2</sup>g<sup>-1</sup> and a pore size of 50 Å. 8 g of this was ground to a particle size of less than 1 µm, cleaned by solvent extraction, sonicated in 200 ml cyclohexane for 20 minutes. 5 mL aliquots were filtered off using a 0.5 µm teflon filter, washed with methanol, dried and stored prior to use. The resin was coated onto denuder sections. Once used the denuder sections were extracted in spectroscopy grade ethyl acetate containing 0.01% triethylamine.

The extract was analysed for nicotine using a nitrogen-phosphorus detector connected to a Shimadzu GC-9A gas chromatograph with a DB-wax 30 inch by 0.32 mm diameter fused silica capillary column with a 1 mL min<sup>-1</sup> helium (primary carrier) flow rate, a 15 mL min<sup>-1</sup> helium (make-up) flow rate, a 4 mL min<sup>-1</sup> hydrogen flow rate and a 75 nL min<sup>-1</sup> air flow rate. Initial column temperature was 175°C, stable for 5 minutes then raised at 0.5°C min<sup>-1</sup> to 180°C. The denuder contained 15.7 microgrammes per m<sup>3</sup> of nicotine. (JC)

#### EQUIPMENT

Comprises an inlet pipe (16) connected to a cyclone (12) Gas is drawn in via the cyclone and fed to denuder sections (22,24,26). These are coated with the resin agglomerate and absorb contaminants from the gas stream. The gas exits via a glass or quartz fabric filter (14) that removed particulates. Once used the denuder sections are treated to release their contaminant load and a gas stream is drawn through them to carry the contaminants to an analysis device, e.g. gas chromatograph.

#### PREFERRED SORBENT

The particle size range is 0.1-7 and most preferably 0.2-4 microns.

#### FILE SEGMENT:

CPI; EPI

#### MANUAL CODE:

CPI: A12-E13; A12-W11D; E07-D03; E07-D04C; E10-J02B4;  
E11-Q03; J01-E02B; J04-C01  
EPI: S03-E09C1; S03-E13C; S03-E14P; S03-E14P1

December 17, 2009

10/536,853

44

L40 ANSWER 13 OF 21 WPIX COPYRIGHT 2009 THOMSON REUTERS on STN  
 ACCESSION NUMBER: 1996-514917 [51] WPIX  
 DOC. NO. CPI: C1996-161216 [51]  
 TITLE: Production of separation reagents used in  
 chromatography - by washing reagents with  
 solution containing amine  
 DERWENT CLASS: A96; B04; J04  
 INVENTOR: FUTAGAWA T  
 PATENT ASSIGNEE: (DAIL-C) DAICEL CHEM IND LTD  
 COUNTRY COUNT: 1

## PATENT INFORMATION:

PATENT NO	KIND	DATE	WEEK	LA	PG	MAIN IPC
JP 08268920	A	19961015	(199651)*	JA	6[0]	
<---						
JP 3905564	B2	20070418	(200728)	JA	9	

## APPLICATION DETAILS:

PATENT NO	KIND	APPLICATION	DATE
JP 3905564	B2	JP 1995-77555	19950403

## APPLICATION DETAILS:

PATENT NO	KIND	APPLICATION	DATE
JP 3905564	B2	JP 1995-77555	19950403

## FILING DETAILS:

PATENT NO	KIND	PATENT NO
JP 3905564	B2 Previous Publ	JP 8268920 A

## INT. PATENT CLASSIF.:

IPC ORIGINAL: C07B0057-00 [I,A]; C07B0057-00 [I,C]; C07C0253-00 [I,C]; C07C0253-34 [I,A]; C07C0255-00 [I,C]; C07C0255-43 [I,A]; C07D0213-00 [I,C]; C07D0213-38 [I,A]

IPC RECLASSIF.: C07B0057-00 [I,A]; C07B0057-00 [I,C]; C07C0253-00 [I,C]; C07C0253-34 [I,A]; C07C0255-00 [I,C]; C07C0255-43 [I,A]; C07D0213-00 [I,C]; C07D0213-38 [I,A]; C07D0279-00 [I,C]; C07D0279-28 [I,A]

## JAP. PATENT CLASSIF.:

MAIN/SEC.: C07B0057-00 310; C07B0057-00 360; C07B0057-00 380; C07C0253-34; C07C0255-43; C07D0213-38; C07D0279-28; G01N0030-88 201 W

## FTERM CLASSIF.:

2G063; 4C036; 4C055; 4H006; 4C055/AA01; 4H006/AA02; 4C036/AA03; 4C036/AA14; 4C036/AA18; 4H006/AD17; 4C055/BA02; 4C055/BA06; 4C055/BA13; 4C055/BA27; 4C055/BB01; 4C055/BB02; 4H006/BB19; 4C055/CA01; 4C055/DA01

## BASIC ABSTRACT:

JP 08268920 A UPAB: 20050514

Production of separation reagents comprising polysaccharide derivs. which are derived from polysaccharides per se containing 3 ppm or more ions, comprises washing the reagents with a solution containing an amine. The ion is one or more ions of chloride, nitrate, phosphate, sulphate, sodium, ammonium,

potassium, calcium and magnesium. The amine is  $\text{NH}_3$ ,  $\text{Me}_2\text{NH}$ ,  $\text{Et}_2\text{NH}$  and  $\text{Et}_3\text{N}$ . The reagents used in separation of optically active isomers.

Optical resolution of chlorpheniramine: separation reagent: amylose tris(3,5-dimethylphenylcarbamate) immobilised on silica gel; column: stainless steel, 25 cm x 0.46 cm; washing with an amine solution: n-hexane/2-propanol/ $\text{Et}_2\text{NH}$  (90/10/0.1 by v/v/v); elution: hexane/2-propanol (90/10), flow rate = 1.0 ml/min. at 25 deg.C; result: resolution rate ( $R_s$ ) = 1.28 (cf.  $R_s$  = 0.43; the column unwashed with the amine solution). The pref. polysaccharides are cellulose, amylose, beta-1,4-chitosan, chitin, beta-1,4-mannan, beta-1,4-xylan, inulin and beta-1,3-glucan; the derivs. are esters and urethanes; mol.weight 1,000-500,000, pref. mol.weight 20,000-500,000; they may be formulated into porous beads of 1 micron - 10 mm in particle size, pref. 1 - 30 micron. They may pref. be immobilised on porous carriers, e.g. polystyrene, polyacrylamide, polyacrylate, silica gel, alumina, magnesia, titanium oxide, glass, silicate, kaolin, at a rate of 1-10- weight%, pref. 5-50 weight%. The washing may be made with a 0.01-50 volume%, pref. 0.01-5 volume%, amine solution in pentane, hexane, heptane, MeOH, EtOH, or 2-PrOH.

USE - The reagents are used in liquid chromatography, where the reagents may be packed in a column directly or as loading material immobilised on carriers. The reagents are partic. effective in separation of optically active substances without tailing.

FILE SEGMENT: CPI  
 MANUAL CODE: CPI: A03-A00A; A12-L04A; B04-C02A; B04-C02E3;  
 B04-C03B; B04-D02; B04-J03A; B05-A01A; B05-A01B;  
 B05-A03A; B05-C01; B05-C02; B07-D04C; B11-B  
 ; B12-M11D; J04-B01B; J04-B01C

L40 ANSWER 14 OF 21 WPIX COPYRIGHT 2009 THOMSON REUTERS on STN  
 ACCESSION NUMBER: 1991-201935 [28] WPIX  
 DOC. NO. CPI: C1991-087363 [21]  
 TITLE: Reversed phase chromatography packing  
 material - comprises silica gel  
 coated with poly:carbo:silane, prepared by treating  
 silica gel particles with  
 poly:silane  
 DERWENT CLASS: A26; A89; J04; P73  
 INVENTOR: KOMIYA K; MORIYAMA H; OHNAKA T; ONAKA T  
 PATENT ASSIGNEE: (TOYJ-C) TOSOH CORP  
 COUNTRY COUNT: 7

#### PATENT INFORMATION:

PATENT NO	KIND	DATE	WEEK	LA	PG	MAIN IPC
EP 436179	A	19910710	(199128)*	EN		
<--						
JP 03233354	A	19911017	(199148)	JA		
<--						
US 5194333	A	19930316	(199313)	EN	5[0]	
<--						
EP 436179	B1	19940330	(199413)	EN	11[0]	
<--						
DE 69007764	E	19940505	(199419)	DE		
<--						
JP 2874297	B2	19990324	(199917)	JA	6	
<--						

#### APPLICATION DETAILS:

PATENT NO	KIND	APPLICATION	DATE
-----------	------	-------------	------

EP 436179 A	EP 1990-124637 19901218
JP 03233354 A	JP 1989-326172 19891218
JP 03233354 A	JP 1990-187282 19900717
JP 2874297 B2	JP 1990-187282 19900717
DE 69007764 E	DE 1990-69007764
19901218	
EP 436179 B1	EP 1990-124637 19901218
DE 69007764 E	EP 1990-124637 19901218
US 5194333 A	US 1990-629625 19901218

## FILING DETAILS:

PATENT NO	KIND	PATENT NO
DE 69007764 E	Based on	EP 436179 A
JP 2874297 B2	Previous Publ	JP 03233354 A

PRIORITY APPLN. INFO: JP 1990-187282 19900717  
 JP 1989-326172 19891218

## INT. PATENT CLASSIF.:

MAIN: B01J0020-32

IPC RECLASSIF.: B01D0015-26 [I,C]; B01D0015-32 [I,A];  
 B01J0020-281 [I,A]; B01J0020-281  
 [I,C]; B01J0020-286 [I,A];  
 B01J0020-30 [I,C]; B01J0020-32  
 [I,A]; G01N0030-00 [I,C]; G01N0030-88 [I,A]  
 B01D0015-32R; B01J0020-32; G01N0030-48A

## ECLA:

## USCLASS NCLM:

## NCLS:

428/405.000  
 210/198.200; 210/656.000; 427/220.000; 428/429.000;  
 428/447.000

## JAP. PATENT CLASSIF.:

## MAIN/SEC.:

## FTERM CLASSIF.:

B01J0020-22 D; G01N0030-48 L  
 2G063; 4G066; 4G066/AA10.D; 4G066/AA22.C;  
 4G066/AB05.D; 4G066/AB18.D; 4G066/AC28.D; 4G066/BA05;  
 4G066/BA20; 4G066/BA23; 4G066/BA28; 4G066/BA38;  
 4G066/CA51; 4G066/DA07; 4G066/EA01; 4G066/FA11;  
 4G066/FA21; 4G066/FA22; 4G066/FA34

## BASIC ABSTRACT:

EP 436179 A UPAB: 20060106

A packing material for reversed phase chromatography comprises silica gel (I) coated with a silicon containing polymer (II). Polymer (II) is pref. a polycarbosilane (IIA) R1 = octyl gp., octadecyl gp. or a phenyl gp. R2 = Methyl, or methylene gp. n = positive integer, but may also be polysilane (IIB) R3 = R1, m = n or a polysiloxane (IIC) R3 = R1 l = n  
 m. The packing material is manufactured by treating the coating on silica gel at a temperature silica -containing-polymer-(II) of 200-500 deg.C in an inert atmosphere of N2, He or Ar. The silica gel used has pore sizes in the range 20-10000 Angstroms.

USE/ADVANTAGE - These reverse phase chromatography packing material have improved durability over prior art. - In an example, a silica gel packing material for reverse phase chromatographic columns was prepared by: Treating porous silica gel particles (5g, 5 micron dia. pore dia. 12 nm) with a polysilane obtained by reacting Na with octadecylmethylsilyl dichloride in toluene and carefully washing and drying the carrier; before heat treating it at 250 deg.C for 120 hrs. The obt'd. material was tested as a packing medium in chromatographic columns and found to be operative and longlasting.

## FILE SEGMENT:

## MANUAL CODE:

CPI; GMPI

CPI: A06-A00E; A12-L04; J04-B01C

December 17, 2009

10/536,853

47

L40 ANSWER 15 OF 21 WPIX COPYRIGHT 2009 THOMSON REUTERS on STN  
 ACCESSION NUMBER: 1991-126600 [18] WPIX  
 DOC. NO. CPI: C1991-054482 [21]  
 TITLE: Silicone polymer coated porous support for  
 column packing - has a durable chemically  
 modified coating and provides high separation ability in  
 liquid chromatography  
 DERWENT CLASS: A26; A89; J04  
 INVENTOR: KANDA T; OHTA T; OHTSU Y; OTA T; OTSU Y; SAKAMOTO A  
 PATENT ASSIGNEE: (SHIS-C) SHISEIDO CO LTD  
 COUNTRY COUNT: 5

## PATENT INFORMATION:

PATENT NO	KIND DATE	WEEK	LA	PG	MAIN IPC
EP 425104	A	19910502 (199118)	* EN		
<--					
JP 03218458	A	19910926 (199145)	JA		
<--					
US 5135649	A	19920804 (199234)	EN	7[0]	
<--					
EP 425104	B1	19931222 (199351)	EN	11[0]	
<--					
DE 69005415	E	19940203 (199406)	DE		
<--					

## APPLICATION DETAILS:

PATENT NO	KIND	APPLICATION	DATE
EP 425104 A		EP 1990-310717	19901001
JP 03218458 A		JP 1989-257288	19891002
JP 03218458 A		JP 1990-83618	19900330
US 5135649 A		US 1990-588044	19900925
DE 69005415 E		DE 1990-69005415	
19901001			
EP 425104 B1		EP 1990-310717	19901001
DE 69005415 E		EP 1990-310717	19901001

## FILING DETAILS:

PATENT NO	KIND	PATENT NO
DE 69005415 E	Based on	EP 425104 A

PRIORITY APPLN. INFO: JP 1990-83618 19900330  
 JP 1989-257288 19891002

## INT. PATENT CLASSIF.:

MAIN: B01J0020-32  
 IPC RECLASSIF.: B01D0015-08 [I,A]; B01D0015-08 [I,C];  
 B01J0020-281 [I,A]; B01J0020-281  
 [I,C]; B01J0020-30 [I,C];  
 B01J0020-32 [I,A]; G01N0030-00 [I,C];  
 G01N0030-88 [I,A]  
 ECLA: B01D0015-08; B01J0020-32  
 USCLASS NCLM: 210/198.200  
 NCLS: 210/502.100; 210/635.000; 210/656.000; 502/401.000;  
 502/402.000; 502/439.000  
 JAP. PATENT CLASSIF.:

MAIN/SEC.: B01J0020-26 L; G01N0030-48 C  
 FTERM CLASSIF.: 2G063; 4G066; 4G066/AA22.C; 4G066/AA32.D;  
 4G066/AA52.D; 4G066/AC28.A; 4G066/AC28.D;  
 4G066/AE19.C; 4G066/BA20; 4G066/BA22; 4G066/BA23;  
 4G066/CA54; 4G066/DA12; 4G066/EA01; 4G066/FA07;  
 4G066/FA17; 4G066/FA21

## BASIC ABSTRACT:

EP 425104 A UPAB: 20050501

A column packing material (I) comprises a porous support (II) coated with a silicone polymer (III) having Si-R and Si-R' bonds where R is a 1-18C hydrocarbon gp. and R' is a hydro hydrophilic gp. having at least one gp. selected from polyoxyalkylene and hydroxyl as a terminal gp.

(II) is a silica gel, alumina, glass bead, zeolite, hydroxyapatite or graphite or a composite powder of synthetic resin coated with a fine inorganic powder. (II) has an average particle size of 2-200 microns, specific surface area 200-800 m<sup>2</sup>/g and a pore size of 40-120 Angstrom. (III) is obtd. from a silicone cpd. of formula

(R'HSiO)<sub>a</sub>(R2R3SiO)<sub>b</sub>(R4R5R6SiO1/2)<sub>c</sub>;

where R1-3 = H (not all at the same time), (halogen subst.) 1-10C hydrocarbon gp.; R4-6 = H or (halogen subst.) 1-10C hydrocarbon gp.; a = 0 or more; b = 0 or more; and c = 0 or 2. When c is 0, the sum of a and b is 3 or more.

USE/ADVANTAGE - (I) is easily produced and has improved durability and high separation ability. Also the eluent pH has a wide range allowing stable and reliable measurements.

FILE SEGMENT: CPI  
 MANUAL CODE: CPI: A06-A00E1; A12-L04; J01-D01A;  
 J04-B01C

L40 ANSWER 16 OF 21 WPIX COPYRIGHT 2009 THOMSON REUTERS on STN  
 ACCESSION NUMBER: 1988-079519 [12] WPIX  
 DOC. NO. CPI: C1988-035623 [21]  
 TITLE: Chromatographic analytical column  
 packing compsn. - comprising non-swelling particles  
 with cation exchange sites carrying hydrophilic,  
 water, film forming resin.

DERWENT CLASS: A91; J04  
 INVENTOR: LANGHORST M A; RANDALL O W; STEVENS T S  
 PATENT ASSIGNEE: (DOWC-C) DOW CHEM CO  
 COUNTRY COUNT: 5

## PATENT INFORMATION:

PATENT NO	KIND	DATE	WEEK	LA	PG	MAIN IPC
GB 2194900	A	19880323	(198812)*	EN	6[1]	
DE 3632404	A	19880407	(198815)#	DE	5	
FR 2604525	A	19880401	(198820)#	FR		
JP 63085450	A	19880415	(198821)#	JA		
DE 3632404	C	19900201	(199005)#	DE		
GB 2194900	B	19910102	(199101)	EN		
CA 1287442	C	19910806	(199136)#	EN		
JP 04064586	B	19921015	(199246)	JA	4	



&lt;--

## APPLICATION DETAILS:

PATENT NO	KIND	APPLICATION	DATE
GB 2194900 A		GB 1986-22065	19860912
GB 2194900 B		GB 1986-22065	19860912
CA 1287442 C		CA 1986-518912	19860924
DE 3632404 A		DE 1986-3632404	19860924
DE 3632404 C		DE 1986-3632404	19860924
FR 2604525 A		FR 1986-13380	19860925
JP 63085450 A		JP 1986-225050	19860925
JP 04064586 B		JP 1986-225050	19860925

## FILING DETAILS:

PATENT NO	KIND	PATENT NO
JP 04064586 B	Based on	JP 63085450 A

PRIORITY APPLN. INFO: GB 1986-22065 19860912  
 CA 1986-518912 19860924  
 DE 1986-3632404 19860924  
 FR 1986-13380 19860925  
 JP 1986-225050 19860925

## INT. PATENT CLASSIF.:

MAIN: G01N0030-48  
 SECONDARY: B01J0041-06  
 MAIN/SEC.: B01J0041-12; G01N0030-96  
 IPC RECLASSIF.: B01D0015-04 [I,A]; B01D0015-04 [I,C]; B01D0015-08 [I,A]; B01D0015-08 [I,C]; B01D0069-00 [I,A]; B01D0069-00 [I,C]; B01D0071-00 [I,C]; B01D0071-06 [I,A]; B01J0020-281 [I,A]; B01J0020-281 [I,C]; B01J0020-285 [I,A]; B01J0020-30 [I,C]; B01J0020-32 [I,A]; B01J0041-00 [I,C]; B01J0041-08 [I,A]; B01J0041-20 [I,A]; B01J0041-20 [I,C]; B01J0041-20 [I,C]; G01N0030-00 [I,C]; G01N0030-02 [I,A]; G01N0030-64 [I,A]; G01N0030-88 [I,A]  
 ECLA: B01J0020-32; B01J0041-20

## ECLA:

## JAP. PATENT CLASSIF.:

MAIN/SEC.: B01D0015-08; B01J0041-06; B01J0041-08 Z; G01N0030-02 B; G01N0030-48 F; G01N0030-48 M; G01N0030-48 P; G01N0030-64 A; G01N0030-88 H

## FTERM CLASSIF.:

2G063; 4D017; 4G067; 4D017/AA01; 4D017/BA11; 4D017/CA14; 4D017/CA17; 4D017/CB01; 4D017/CB10; 4D017/DA03; 4D017/EB02; 4D017/EB03

## BASIC ABSTRACT:

GB 2194900 A UPAB: 20050427

A chromatographic analytical column contains an anion-exchange packing comprising (a) a substrate of non-swelling particles with cation-exchanging sites at least on the available surfaces, and with dia. 1-75 microns, and (b) a chromatographically active anion-exchange liquid coating irreversibly attached to the surfaces of the particles. The coating is a hydrophilic, water-soluble, film-forming aminated resin having anion-exchanging sites which attract available cation-exchanging sites on the particles, so that the liquid coating is retained by electrostatic bonds on the available surfaces of the particles.

The particles are pref. monodisperse, with dia. 3-20 (4-10) microns, and may be of insol. synthetic resin, especially of the gel type, or may be SiO<sub>2</sub> or glass beads. The liquid coating is especially a water-soluble aminated poly(vinylaromatic) resin.

USE - Chromatographic separation of anions comprises passing a liquid solution containing the anions through the bed of chromatographic packing, and eluting the anions from the bed. When the particles are of SiO<sub>2</sub> or glass, the pH of the eluent is less than 8. (claimed).

FILE SEGMENT: CPI  
MANUAL CODE: CPI: A12-L04; A12-M; J04-B01C

L40 ANSWER 17 OF 21 WPIX COPYRIGHT 2009 THOMSON REUTERS on STN  
ACCESSION NUMBER: 1987-087995 [13] WPIX  
DOC. NO. CPI: C1987-036456 [21]  
TITLE: Spherical non-porous  
silica particles - prepared by hydrolytic  
polycondensation of tetra:alkoxy-silane(s) in two  
stages to control the particle size  
DERWENT CLASS: E36; J04  
INVENTOR: GIESCHE H; KINKEL J; UNGER K  
PATENT ASSIGNEE: (MERE-C) MERCK PATENT GMBH  
COUNTRY COUNT: 13

## PATENT INFORMATION:

PATENT NO	KIND	DATE	WEEK	LA	PG	MAIN IPC
EP 216278	A	19870401	(198713)*	DE	8[0]	
<--						
DE 3534143	A	19870402	(198714)	DE		
<--						
AU 8662469	A	19870326	(198719)	EN		
<--						
JP 62072514	A	19870403	(198719)	JA		
<--						
DE 3616133	A	19871119	(198747)	DE		
<--						
CN 86106689	A	19870527	(198833)	ZH		
<--						
US 4775520	A	19881004	(198842)	EN	6	
<--						
US 4911903	A	19900327	(199018)	EN		
<--						
CA 1280399	C	19910219	(199113)	EN		
<--						
EP 216278	B	19920304	(199210)	EN		
<--						
DE 3684071	G	19920409	(199216)	DE		
<--						
JP 08025739	B2	19960313	(199615)	JA	6[0]	
<--						

## APPLICATION DETAILS:

PATENT NO	KIND	APPLICATION	DATE
EP 216278 A		EP 1986-112677	19860913
DE 3534143 A		DE 1985-3534143	19850925
DE 3616133 A		DE 1985-3534143	19850925
DE 3684071 G		DE 1985-3534143	19850925

DE 3534143 A  
DE 3616133 A  
DE 3684071 G  
JP 62072514 A  
JP 08025739 B2  
US 4775520 A  
US 4911903 A

DE 1986-3616133 19860514  
DE 1986-3616133 19860514  
DE 1986-3616133 19860514  
JP 1986-225082 19860925  
JP 1986-225082 19860925  
US 1986-911534 19860925  
US 1988-218000 19880712

## FILING DETAILS:

PATENT NO	KIND	PATENT NO
JP 08025739 B2	Based on	JP 62072514 A

PRIORITY APPLN. INFO: DE 1986-3616133 19860514  
DE 1985-3534143 19850925

## INT. PATENT CLASSIF.:

MAIN: C01B0033-18  
SECONDARY: G01N0030-48  
MAIN/SEC.: C07K0003-20; C12N0009-00; G01N0015-00  
IPC RECLASSIF.: B01D0015-08 [I,A]; B01D0015-08 [I,C]; B01D0015-26 [I,C]; B01D0015-32 [I,A]; B01J0020-10 [I,A]; B01J0020-10 [I,C]; B01J0020-28 [I,A]; B01J0020-28 [I,C]; B01J0020-281 [I,A]; B01J0020-281 [I,C]; B01J0020-283 [I,A]; B01J0020-30 [I,C]; B01J0020-32 [I,A]; C01B0033-00 [I,C]; C01B0033-12 [I,A]; C01B0033-16 [I,A]; C01B0033-18 [I,A]; G01N0030-00 [I,C]; G01N0030-88 [I,A]; B01D0015-32R; B01J0020-10B; B01J0020-32; C01B0033-16B; C01B0033-18

## ECLA:

M01P0004:32; M01P0004:52; M01P0004:61; M01P0004:62; M01P0004:64; M01P0006:12; Y01N0006:00  
423/335.000

## USCLASS NCLM:

NCLS: 423/338.000; 423/339.000; 502/008.000; 556/455.000

## JAP. PATENT CLASSIF.:

MAIN/SEC.: B01D0015-08; B01J0020-02 D; B01J0020-10; B01J0020-10 A; C01B0033-12; C01B0033-18 Z; C08G0077-06; G01N0030-48 C; G01N0030-48 G; G01N0030-48 K; G01N0030-88 101 K; G01N0030-88 201 G; G01N0030-88 201 X

## FTERM CLASSIF.:

2G063; 4D017; 4G066; 4G072; 4J246; 4D017/AA09; 4G066/AA14.D; 4G066/AA22.B; 4G072/AA25; 4G072/AA28; 4G066/AA52.D; 4G066/AB06.A; 4G066/AB06.D; 4G066/AB13.A; 4G066/AB18.A; 4D017/BA03; 4G066/BA09; 4G066/BA20; 4G066/BA26; 4G072/BB05; 4G072/BB07; 4D017/CA05; 4D017/CA14; 4G066/CA20; 4G066/CA54; 4G066/CA56; 4D017/CB01; 4G072/CC02; 4G072/CC05; 4G072/CC10; 4D017/DB03; 4G072/DD04; 4G072/DD05; 4G072/DD06; 4G066/EA01; 4D017/EB02; 4G066/FA03; 4G066/FA21; 4G066/FA34; 4G072/GG01; 4G072/GG03; 4G072/HH30; 4G072/JJ11; 4G072/JJ23; 4G072/JJ38; 4G072/KK03; 4G072/KK13; 4G072/LL09; 4G072/LL11; 4G072/MM02; 4G072/MM03; 4G072/MM31; 4G072/PP01; 4G072/PP03; 4G072/QQ16; 4G072/RR05; 4G072/RR12; 4G072/TT01; 4G072/UU11; 4G072/UU13

## BASIC ABSTRACT:

EP 216278 A UPAB: 20060105

Spherical silica particles (I) are produced by hydrolytic polycondensation of tetraalkoxysilanes (II) in aqueous alcoholic ammonia. The process involves the initial production of a soluble of prim. particles, followed by the addition of further (II) in a continuous and controlled way, to obtain highly-monomodisperse, non-porous particles of mean dia. 0.05-10 microns with a standard deviation of not more than 5%. (I) contains covalently-bound organic gps. in the matrix, which are normally used for the modification of silica gel.

The polycondensation is carried out at 35-75 (pref. 40-65) deg.C; (II) is derived from 1-3C alcohol (especially EtOH); 0.1-100 (pref. 1-30) weight% of (II) is replaced with organo-tri-alkoxysilane.

USE/ADVANTAGE - (I) are useful as sorption materials, especially for the reversed-phase chromatography of proteins (claimed). The characteristics of the particles (see above) make (I) especially suitable for this application w.r.t. prior-art materials, and also enable higher column efficiencies and shorter analysis times.

FILE SEGMENT: CPI  
MANUAL CODE: CPI: E31-P01; J01-D01A; J04-B01C

L40 ANSWER 18 OF 21 WPX COPYRIGHT 2009 THOMSON REUTERS on STN  
ACCESSION NUMBER: 1985-191764 [32] WPX  
DOC. NO. CPI: C1985-083642 [21]  
DOC. NO. NON-CPI: N1985-143899 [21]  
TITLE: Separation of chemical substances especially mixts. of optical

isomers - by using nitrate of poly:saccharide especially in chromatographic column.  
DERWENT CLASS: A89; B05; C03; J04; S03  
INVENTOR: OKAMOTO I; SHIBATA T  
PATENT ASSIGNEE: (DAIC-C) DAICEL CHEM IND LTD; (DAIC-C) DAINICHISEIKA COLOR & CHEM MFG  
COUNTRY COUNT: 9

## PATENT INFORMATION:

PATENT NO	KIND	DATE	WEEK	LA	PG	MAIN IPC
EP 150849	A	19850807	(198532)*	EN	19	[0]
JP 60161928	A	19850823	(198540)	JA		
US 4714555	A	19871222	(198801)	EN		
EP 150849	B	19901031	(199044)	EN		[0]
DE 3580286	G	19901206	(199050)	DE		
JP 04075210	B	19921130	(199252)	JA	0	

## APPLICATION DETAILS:

PATENT NO	KIND	APPLICATION	DATE
EP 150849 A		EP 1985-100948	19850130
JP 60161928 A		JP 1984-15760	19840131
JP 04075210 B		JP 1984-15760	19840131
US 4714555 A		US 1987-24877	19870311

## FILING DETAILS:

PATENT NO	KIND	PATENT NO
JP 04075210 B	Based on	JP 60161928 A
PRIORITY APPLN. INFO: JP 1984-15760 19840131		
INT. PATENT CLASSIF.:		
MAIN:	C07B0063-00	
SECONDARY:	G01N0030-48	
IPC RECLASSIF.:	B01J0020-22 [I,C]; B01J0020-24 [I,A]; B01J0020-281 [I,C]; B01J0020-291 [I,A]; C07B0031-00 [I,A]; C07B0031-00 [I,C]; C07B0057-00 [I,A]; C07B0057-00 [I,C]; C07B0063-00 [I,A]; C07B0063-00 [I,C]; C07C0045-00 [I,C]; C07C0045-78 [I,A]; C07C0067-00 [I,A]; C07C0067-00 [I,C]; C08B0005-00 [I,C]; C08B0005-04 [I,A]; G01N0030-00 [I,C]; G01N0030-88 [I,A]; C07C0045-78; C07C0045-78M; C08B0005-04	
ECLA:		
JAP. PATENT CLASSIF.:		
MAIN/SEC.:	B01J0020-24 C; B60K0017-04 G; B60K0041-00 301 A; B60K0041-00 301 B; B60K0041-00 301 C; B60K0041-00 301 D; B60K0041-00 301 L; B60K0041-22; B60K0041-28; B60K0006-04; B60K0006-04 150; B60K0006-04 151; B60K0006-04 170; B60K0006-04 350; B60K0006-04 360; B60K0006-04 400; B60K0006-04 530; B60K0006-04 733; B60K0006-20 310; B60K0006-20 320; B60K0006-20 350; B60K0006-20 360; B60K0006-20 380; B60K0006-20 400; B60K0006-36; B60K0006-365; B60K0006-387; B60K0006-40; B60K0006-48; B60K0006-547; B60K0009-00 C; B60K0009-00 Z; B60L0011-14; C07B0031-00 X; C07B0057-00; C07B0057-00 310; C07B0063-00; C07B0063-00 F; C07C0067-00 X; F16D0025-14 640 D; F16D0025-14 640 F; F16D0025-14 640 U; F16H0061-02; F16H0061-08; F16H0063-46; G01N0030-48 W; B60K0006-04 (ZHV); B60K0006-36 (ZHV)	
INDEX:	F16H0103:12; F16H0059:08; F16H0059:18; F16H0059:72	
FTERM CLASSIF.:	2G063; 3D035; 3D039; 3D041; 3D202; 3J052; 3J057; 3J552; 4G066; 4H006; 4H040; 5H111; 5H115; 3J052/AA01; 3D039/AA02; 4H006/AA03; 4H040/AA03; 3D039/AA04; 4G066/AA22.C; 3D041/AA53; 3D039/AB01; 3D039/AB27; 3D041/AC00; 4G066/AC02.B; 3D041/AC02; 3D039/AC03; 3D041/AC06; 3D041/AC08; 3D041/AC15; 3D039/AC39; 4H006/AC83; 4H040/AC83; 3D041/AD02; 4G066/AD06.B; 3D041/AD17; 4H006/AD17; 4H040/AD17; 3D041/AD22; 3D041/AD23; 3D041/AD31; 3D041/AD32; 3D041/AD35; 3D041/AD39; 3D041/AD41; 3D041/AD51; 3D041/AE16; 3D041/AE22; 3D041/AE32; 3D041/AE33; 3D041/AF00; 3J057/BA04; 3J052/CA02; 3J052/CA03; 4G066/CA19; 4G066/DA07; 4G066/EA01; 3J052/EA03; 3J052/FB25; 3J052/FB33; 3J057/GA66; 3J057/GB27; 3J057/GC06; 3J057/GC09; 3J052/GC73; 3J057/GD08; 3J057/GD20; 3J057/GE07; 3J052/HA02; 3J057/HH01; 3J057/JJ01; 3J057/JJ04; 3J057/JJ06; 3J052/KA01; 3J052/LA01; 3J552/MA02; 3J552/MA12; 3J552/MA13; 3J552/MA14; 3J552/MA17; 3J552/MA22; 3J552/MA26; 3J552/NA01; 3J552/NB01; 3J552/NB04; 3J552/NB06; 3J552/NB09; 3J552/PA03; 3J552/PA20; 3J552/PA51; 3J552/PA52; 5H115/PG04; 5H115/PI16; 5H115/PI22; 5H115/PI29; 5H115/PI30; 5H115/PO17; 5H115/PU02; 5H115/PU10;	

5H115/PU22; 5H115/PU24; 5H115/PU25; 5H115/QA01;  
 5H115/QA10; 3J552/QA30.B; 3J552/QA30.C; 5H115/QE10;  
 5H115/QI04; 5H115/QI09; 5H115/QN03; 3J552/RA20;  
 3J552/RA29; 3J552/RA30; 5H115/RB08; 3J552/RB17;  
 5H115/RE05; 5H115/RE20; 3J552/SA03; 3J552/SA07;  
 3J552/SA15; 3J552/SB33; 5H115/SE04; 5H115/SE05;  
 5H115/SE08; 5H115/SJ12; 5H115/SJ13; 5H115/TB01;  
 5H115/TE02; 5H115/TE08; 5H115/TI01; 5H115/TO02;  
 5H115/TO05; 5H115/TO21; 5H115/TO23; 5H115/TO30;  
 3J552/UA03; 3J552/UA09; 3J552/UA10; 3J552/VA32.Z;  
 3J552/VA37.Z; 3J552/VA42.Z; 3J552/VA48.W;  
 3J552/VA62.Z; 3J552/VA63.Z; 3J552/VA64.Z;  
 3J552/VA65.Z; 3J552/VA66.Z; 3J552/VA70.Z;  
 3J552/VA76.Z; 3J552/VB01.Z; 3J552/VB04.Z;  
 3J552/VB10.Z; 3J552/VC01.Z; 3J552/VC07.Z;  
 3J552/VC09.Z; 3J552/VC10.Z; 3J552/VD02.Z;  
 3J552/VD11.Z; 3J552/VD17.Z

## BASIC ABSTRACT:

EP 150849 A UPAB: 20050423

Separation of a chemical substance (I) from a mixture containing it comprises treatment with a nitrate or nitrate gp-containing derivative of apolysaccharide. (I) is especially an optical isomer present in a mixture of optical isomers.

The polysaccharide is synthetic, natural or modified natural material, especially cellulose. It has a number average polymerisation degree of 3-5000. The nitrate and nitrate gp.-containing derivative has a nitration ratio of 0.3-1, and the particle size is 1 micron to 10 mm. These particles may be deposited on a carrier at 1-100 weight % based on the weight of carrier. The carrier may be silica gel of particle size 1 micron - 10 mm with a pore size of 10 Angstroms - 100microns. The procedure may involve use of the cellulose derivative in a chromatographic column or layer.

Seps. can be made directly, especially for optical isomer mixts. even when they could not previously be resolved directly. Such resolutions are useful in the preparation of medicinal agents, pesticides etc.

## FILE SEGMENT:

CPI; EPI

## MANUAL CODE:

CPI: A03-A03; A03-A05; A12-L04; B04-C02;

B11-B; C04-C02; C11-B;

J01-D01A

EPI: S03-E09C

L40 ANSWER 19 OF 21

WPIX COPYRIGHT 2009

THOMSON REUTERS ON STN

ACCESSION NUMBER:

1985-146198 [24] WPIX

DOC. NO. CPI:

C1985-063600 [21]

TITLE:

High performance columns for anion analysis  
 - containing pellicular packing of cation resin  
 beads coated with anion resin microparticles

DERWENT CLASS:

A13; A89; J04

INVENTOR:

LANGHORST M A; STEVENS T S

PATENT ASSIGNEE:

(DOWC-C) DOW CHEM CO

COUNTRY COUNT:

1

## PATENT INFORMATION:

PATENT NO	KIND DATE	WEEK	LA	PG	MAIN IPC
US 4519905	A	19850528	(198524)*	EN	11[3]

&lt;--

## APPLICATION DETAILS:

PATENT NO	KIND	APPLICATION	DATE
US 4519905 A		US 1981-234520	19810217
US 4519905 A		US 1982-431431	19820930
US 4519905 A		US 1984-626569	19840702

PRIORITY APPLN. INFO: US 1984-626569 19840702  
 US 1981-234520 19810217  
 US 1982-431431 19820930

## INT. PATENT CLASSIF.:

IPC RECLASSIF.: B01D0015-08 [I,A]; B01D0015-08 [I,C]; B01J0041-20 [I,A]; B01J0041-20 [I,C]; G01N0030-00 [I,C]; G01N0030-56 [I,A]; G01N0030-96 [I,A]  
 B01J0020-286; B01J0041-20; G01N0030-56; G01N0030-96  
 L01J0220:20

ECLA:

ICO:

USCLASS NCLM:

NCLS:

210/198.200  
 210/635.000; 521/028.000

## BASIC ABSTRACT:

US 4519905 A UPAB: 20050423

An improved chromatographic analytical column contains anion-exchange particles obtd. by agglomerating a monolayer of microparticles (I) of insoluble synthetic resin having anion-exchange sites at least on the outer surfaces (and pref. throughout) onto a pressure-packed bed of substrate particles (II) of insoluble synthetic resin having cation-exchange sites at least on their available surfaces. (II) are of lower porosity relative to (I). (I) have volume average dia. (D) 50-100 (pref. 50-600 and especially 50-300) Angstroms and are pref. monodisperse. (II) have D=5-75 (more pref. 5-35 and especially 5-20) microns and are pref. also monodisperse (i.e. 90-95% of particles are within the range 0.5-1.5 (pref. 0.67-1.33) D) and of the gel type.

A wide variety of addition d condensation backbone polymers may be used for (I) and (II); crosslinked poly(vinyl aromatic) resins are pref. Especially suitable for (II) are styrene-divinylbenzene copolymer resins sulphonated e.g. as described in US3,966,596. (I) are prepared as latex particles using a comonomer which can be aminated or quaternised.

USE/ADVANTAGE - The column packing is stable and provides high performance in the analysis of anions compared with prior art pellicular columns; it may be used in strongly basic media, unlike silica-based columns.

FILE SEGMENT:

CPI

MANUAL CODE:

CPI: A12-L04; A12-M; J04-B01C

L40 ANSWER 20 OF 21

WPIX COPYRIGHT 2009

THOMSON REUTERS on STN

ACCESSION NUMBER:

1984-259782 [42] WPIX

DOC. NO. CPI:

C1984-110034 [21]

DOC. NO. NON-CPI:

N1984-194023 [21]

TITLE:

Molecular sieve type liquid chromatograph  
 - includes multi-passage valve and sample feeding and collecting devices

DERWENT CLASS:

J04

INVENTOR:

SUZUKI N; TAKE T

PATENT ASSIGNEE:

(SHMC-C) SHIMIZU CONSTRUCTION

COUNTRY COUNT:

1

## PATENT INFORMATION:

PATENT NO	KIND	DATE	WEEK	LA	PG	MAIN IPC
JP 59157563	A	19840906	(198442)* JA	9	[6]	

&lt;--

## APPLICATION DETAILS:

PATENT NO	KIND	APPLICATION	DATE
JP 59157563 A		JP 1983-32278	19830228

PRIORITY APPLN. INFO: JP 1983-32278 19830228

INT. PATENT CLASSIF.:  
 IPC RECLASSIF.: G01N0030-00 [I,C]; G01N0030-06 [I,A]; G01N0030-14 [I,A]; G01N0030-46 [I,A]; G01N0030-88 [I,A]  
 ECLA: G01N0030-06  
 JAP. PATENT CLASSIF.:  
 MAIN/SEC.: G01N0030-14 A; G01N0030-46 G; G01N0030-46 Z; G01N0030-88 H  
 FTERM CLASSIF.: 2G063  
 BASIC ABSTRACT:

JP 59157563 A UPAB: 20050421

Chromatograph operation includes connecting a sample-feeding device to the spout side of a feed pump for feeding an eluent under pressure, connecting an exchange valve, which has an introducing part and a number of outlet parts and can open selectively the path between the introducing part and the optional outlet part, to the outlet side of the sample-introducing device, connecting each introducing end of a precolumn packed with porous glass beads (80-120 mesh, about 3000 angstrom) to at least two outlet parts of the exchange valve. The outlet part of each precolumn is connected to each introducing part of an exchange valve which has a number of introducing parts and an outlet part and can open selectively the path between the outlet part and optional introducing part. A molecular sieve type separation column packed with porous glass beads or porous silica gel is connected to the outlet part of the exchange valve, and a detection means is connected to the outlet side of the molecular sieve type separation column.

ADVANTAGE - The chromatograph is suitable for the analysis of a sample containing suspended substance in which air is opt. occluded. As the suspended substance is removed by the precolumn, no pretreatment of sample is required. Since several precolumns are provided in the system, many determinations can be carried out continuously by exchanging the precolumns in order.

FILE SEGMENT: CPI  
 MANUAL CODE: CPI: J04-E01C

L40 ANSWER 21 OF 21 WPIX COPYRIGHT 2009 THOMSON REUTERS on STN  
 ACCESSION NUMBER: 1982-89198E [42] WPIX  
 TITLE: Adsorbent used for packing liquid chromatography column - obtd. by coating synthetic resin on porous particles  
 DERWENT CLASS: A89; J01  
 INVENTOR: KONDO Y  
 PATENT ASSIGNEE: (DAICEL-C) DAICEL CHEM INDS LTD  
 COUNTRY COUNT: 1

## PATENT INFORMATION:

PATENT NO	KIND	DATE	WEEK	LA	PG	MAIN IPC
JP 57147434	A	19820911	(198242)*	JA	3	

<--

## APPLICATION DETAILS:

PATENT NO	KIND	APPLICATION	DATE
-----------	------	-------------	------



JP 57147434 A

JP 1981-33087 19810310

PRIORITY APPLN. INFO: JP 1981-33087

19810310

INT. PATENT CLASSIF.:

IPC RECLASSIF.:

B01J0020-22 [I,C]; B01J0020-26  
[I,A]; B01J0020-28 [I,A];  
B01J0020-28 [I,C]; B01J0020-281  
[I,A]; B01J0020-281 [I,C];  
B01J0020-291 [I,A]; C07C0001-00 [I,A];  
C07C0001-00 [I,C]; C07C0067-00 [I,A]; C07C0067-00  
[I,C]; C07C0007-00 [I,C]; C07C0007-12 [I,A];  
G01N0030-00 [I,C]; G01N0030-88 [I,A]

## BASIC ABSTRACT:

JP 57147434 A UPAB: 20050420

Adsorbing agent is obtd. by coating a thermoplastic system synthetic resin on a completely porous type minute particle having 10 angstrom average pore size. The particle consists of silica gel, alumina, diatomaceous earth, or glass beads. The resin is polyethylene, polypropylene, polyester, PET, polyamide, nylon, PMMA, PVC or polystyrene.

Completely porous silica gel having 10 micron average particle size and 500 angstrom average pore size was treated with diphenyl dichlorosilane and triethylamine. The resulting treated silica gel was immersed in THF solution of triphenyl methyl polymethacrylate for 30 minutes, separated and dried to obtain an adsorbing agent used for separation process. The agent was packed in a liquid chromatography column, and used in separation of acetone, benzene, or toluene.

FILE SEGMENT:

CPI

MANUAL CODE:

CPI: A11-B05; A12-L04; J01-D01A

=&gt; D L41 1-4 IFULL

L41 ANSWER 1 OF 4 WPIX COPYRIGHT 2009 THOMSON REUTERS on STN

ACCESSION NUMBER: 2001-648288 [74] WPIX

CROSS REFERENCE: 1998-386901; 1998-506688; 1999-009395; 1999-045150;  
1999-070314; 1999-105524; 1999-167458; 1999-190174;  
1999-277659; 1999-287986; 1999-313210; 1999-457857;  
2000-349558; 2000-375583; 2000-557659; 2000-678760;  
2001-071577; 2001-244716; 2001-381916; 2001-463931;  
2001-548990; 2001-611309; 2001-637908; 2002-055352;  
2002-065549; 2002-082150; 2002-204872; 2002-314786;  
2002-424420; 2002-424729; 2002-566535; 2002-689463;  
2002-690602; 2003-017190; 2003-093162; 2003-110192;  
2003-196844; 2003-218954; 2003-310425; 2003-615241;  
2003-706911; 2003-743830; 2003-777165; 2004-040958;  
2004-051045; 2004-106502; 2004-118797

DOC. NO. CPI:

C2001-191238 [74]

TITLE:

Matched ion polynucleotide chromatography  
for separating a mixture of RNA molecules involves  
the use of non-polar reverse phase media, counterion  
agent and an organic solvent to release the  
polynucleotides from the media

DERWENT CLASS:

A89; B04; D16

INVENTOR:

AZARANI A; HECKER K H; HORNBY D; MATIN M; TAYLOR P D

PATENT ASSIGNEE:

(AZAR-I) AZARANI A; (HECK-I) HECKER K H; (HORN-I)  
HORNBY D; (MATI-I) MATIN M; (TAYL-I) TAYLOR P D;  
(TRAN-N) TRANSGENOMIC INC

COUNTRY COUNT:

92

PATENT INFORMATION:

PATENT NO	KIND	DATE	WEEK	LA	PG	MAIN IPC
WO 2001066218	A1	20010913	(200174)*	EN	71[21]	
<---						
AU 2001047347	A	20010917	(200204)	EN		
<---						
US 20010051715	A1	20011213	(200204)	EN		
<---						

## APPLICATION DETAILS:

PATENT NO	KIND	APPLICATION	DATE
WO 2001066218 A1		WO 2001-057604	20010309
US 20010051715 A1	Provisional	US 2000-187974P	20000309
US 20010051715 A1	Provisional	US 2000-213948P	20000623
AU 2001047347 A		AU 2001-47347	20010309
US 20010051715 A1		US 2001-802466	20010309

## FILING DETAILS:

PATENT NO	KIND	PATENT NO
AU 2001047347 A	Based on	WO 2001066218 A

PRIORITY APPLN. INFO: US 2000-213948P 20000623  
 US 2000-187974P 20000309  
 US 2001-802466 20010309

## INT. PATENT CLASSIF.:

IPC RECLASSIF.: B01D0015-26 [I,C]; B01D0015-36 [I,A]

ECLA: B01D0015-36P

USCLASS NCLM: 536/025.400

## BASIC ABSTRACT:

WO 2001066218 A1 UPAB: 20050902

NOVELTY - Stabilization of RNA molecule, (1) against degradation comprising degrading the catalyzing agent to a separation medium having a non-polar separation surface in the presence of counterion agent and eluting and collecting an eluant containing (1), free of catalyzing agent, is new.

DETAILED DESCRIPTION - Stabilization of RNA molecule (1) against degradation involves:

(a) applying a solution of (1) and degradation catalyzing agent to a separation medium having a non-polar separation surface in the presence of counterion agent;

(b) eluting (1) from the medium by passing a mobile phase having an organic solvent through the medium, under conditions for separation of (1) from the catalyzing agent; and

(c) collecting an eluant containing (1), free of catalyzing agent.

USE - The method is useful for stabilizing the RNA molecule against degradation and to segregate RNA molecules above a lengths exceeding about 200nt and to segregate tRNA from rRNA and tRNA and rRNA from mRNA molecules.

ADVANTAGE - The MIPC enhances the purification and stabilization of RNA. The method is fast (10 - 30 minutes), safe, reliable, convenient, reproducible and quantitative. The method minimizes use of toxic and reactive chemicals. MIPC segregation of RNA provides intact RNA molecules or size range of RNA molecules and avoiding chemical covalent modification which can occur using the prior art gel based separation methods. The shorter processing time required, thus the method decrease the chance for RNA degradation to occur.

## TECHNOLOGY FOCUS:

BIOTECHNOLOGY - Preferred Agent: The catalyzing agent is an

enzyme (preferably nuclease, especially an RNase).

BIOLOGY - Preferred Method: (1) is separated from the catalyzing agent by Matched Ion Polynucleotide Chromatography (MIPC) in a batch process, under conditions, such that the secondary structure of (1) is substantially denatured and at at least 50 (preferably at least 70) degrees C. The mRNA molecule is substantially denatured by using a chemical reagent. Separation medium and separation conditions are substantially free of multivalent cations capable of interfering with polynucleotide separations. A stabilized solution of (1) is substantially free of RNase and devoid of RNase inhibitors and stable at room temperature.

POLYMERS - Preferred Medium: Separation medium comprises particles of insoluble polysaccharide. The separation surface of the particle is coated with a hydrocarbon or non-polar hydrocarbon substituted polymer or have substantially all polar groups reacted with the non-polar or substituted hydrocarbon groups. The surfaces are non-polar. The medium comprises the polymer beads of an average diameter of 0.5 - 100 microns. The beads are optionally substituted beads with a moiety selected from 1 - 1000000C hydrocarbon (Preferably 18C alkylated nonporous poly(styrene-divinylbenzene) polymer beads, especially monolith). The medium is prepared by using reagents and conditions that are substantially free of multivalent cations. The medium is subjected to acid wash treatment to remove any residue surface metal contaminants and to treatment with a multivalent cation - binding agent.

INORGANIC CHEMISTRY - Preferred Medium: The separation medium comprises particles selected from silica, silica carbide, silica nitrite, titanium oxide, aluminum oxide, zirconium oxide or diatomaceous earth. The counterion agent is a quaternary ammonium salt.

ORGANIC CHEMISTRY - Preferred Components: The particles of the medium are carbon. The organic solvent is alcohol, nitrile ester and/or ether. The counterion agent is lower alkyl primary, secondary or tertiary amine and/or lower trialkylammonium salt.

#### EXTENSION ABSTRACT:

SPECIFIC COMPOUNDS - Dimethylformamide, tetrahydrofuran, acetonitrile are specifically claimed as the organic solvents. Octylammonium acetate, Octadimethylammonium acetate, decylammonium acetate, octadecylammonium acetate, pyridiniumammonium acetate, cyclohexylammonium acetate, diethylammonium acetate, propylethylammonium acetate, propyldiethylammonium acetate, butylethylammonium acetate, methylhexyl ammonium acetate, tetramethylammonium acetate, tetraethylammonium acetate, tetrabutylammonium acetate, tetrabutylammonium acetate, dimethyldiethylammonium acetate, triethylammonium acetate, tripropylammonium acetate, tributylammonium acetate, tetrapropylammonium acetate, tetrabutylammonium acetate, triethylammonium hexafluoroisopropyl alcohol and tetrabutylammonium bromide are specifically claimed as a counterion agent.

EXAMPLE - A Matched Ion Polynucleotide Chromatography (MIPC) analysis of a 0.16 - 1.77 Kb RNA ladder was performed using octadecyl modified, non-porous poly(ethyvinylbenzene-divinylbenzene) beads packed in DNASEP cartridge (RTM; 50x7.8 mm ID reverse phase column) and using WAVE(RTM; nucleic acid fragment analysis system). Buffer A was 0.1M TEAA, pH 7, and buffer B was 0.1 TEAA (triethylammonium acetate), 25 v/v.% acetonitrile, pH 7. At time (minutes) 0, 1, 16, 22, 22.5, 23, 24, 25 and 27, % buffer B were 38, 40, 60, 66, 70, 100, 100, 38 and 38 respectively. The flow rate was 0.9 ml/min and the

column temperature was 75degreesC. Ultraviolet detection was performed at 260nm. The injection volume was 5mul. The obtained sample contained a mixture of 8 RNAs having lengths of 155, 280, 400, 530, 780, 1280, 1520 and 1770 nucleotides, segregated. Prior to the injection, the column was equilibrated with 75% acetonitrile for 30 - 45 minutes. The column was then equilibrated using 38% buffer B for 30 minutes. Prior to the elution of RNA, two control gradient elutions (using the same gradient conditions as for the RNA) were performed a first injection of 0.5mM ethylenediaminetetraacetate (EDTA) (10 mul) and a second injection of nuclease free water (10 mul).

FILE SEGMENT: CPI  
 MANUAL CODE: CPI: A12-L04A; B04-E02; B11-C01; B11-C08; D05-H12A; D05-H18

L41 ANSWER 2 OF 4 WPIX COPYRIGHT 2009 THOMSON REUTERS on STN  
 ACCESSION NUMBER: 2001-343460 [36] WPIX  
 DOC. NO. CPI: C2001-106319 [36]  
 TITLE: New oxyiminoalkanoic acid derivative crystals, useful for treating e.g. diabetes, hyperlipidemia, impaired glucose tolerance, inflammatory diseases and arteriosclerosis  
 DERWENT CLASS: B03  
 INVENTOR: IMOTO H; MOMOSE Y  
 PATENT ASSIGNEE: (TAKE-C) TAKEDA CHEM IND LTD; (TAKE-C) TAKEDA YAKUHHIN KOGYO KK  
 COUNTRY COUNT: 92

## PATENT INFORMATION:

PATENT NO	KIND	DATE	WEEK	LA	PG	MAIN IPC
WO 2001032637	A1	20010510	(200136)*	EN	48[3]	
JP 2001192375	A	20010717	(200144)	JA	18	
AU 2000079581	A	20010514	(200149)	EN		
KR 2001102429	A	20011115	(200231)	KO		
JP 2002097139	A	20020402	(200238)	JA	17	
NO 2002002007	A	20020624	(200253)	NO		
EP 1224178	A1	20020724	(200256)	EN		
HU 2002003884	A2	20030328	(200333)	HU		
CN 1407975	A	20030402	(200345)	ZH		
EP 1224178	B1	20030625	(200349)	EN		
DE 60003577	E	20030731	(200357)	DE		
TW 558554	A	20031021	(200424)	ZH		
US 6777435	B1	20040817	(200454)	EN		
KR 433885	B	20040604	(200465)	KO		

## APPLICATION DETAILS:

PATENT NO	KIND	APPLICATION	DATE
WO 2001032637	A1	WO 2000-JP7482	20001026
TW 558554	A	TW 2000-122215	20001023

AU 2000079581 A  
 CN 1407975 A  
 DE 60003577 E  
 20001026  
 EP 1224178 A1  
 EP 1224178 B1  
 DE 60003577 E  
 NO 2002002007 A  
 EP 1224178 A1  
 HU 2002003884 A2  
 EP 1224178 B1  
 DE 60003577 E  
 US 6777435 B1  
 KR 433885 B  
 JP 2001192375 A  
 JP 2002097139 A Div Ex  
 JP 2002097139 A  
 KR 2001102429 A  
 KR 433885 B  
 HU 2002003884 A2  
 US 6777435 B1  
 NO 2002002007 A

AU 2000-79581 20001026  
 CN 2000-816789 20001026  
 DE 2000-60003577  
 EP 2000-970081 20001026  
 EP 2000-970081 20001026  
 EP 2000-970081 20001026  
 WO 2000-JF7482 20001026  
 WO 2000-JF7482 20001026  
 WO 2000-JF7482 20001026  
 WO 2000-JF7482 20001026  
 WO 2000-JF7482 20001026  
 WO 2000-JF7482 20001026  
 WO 2000-JF7482 20001026  
 WO 2000-JF7482 20001026  
 JF 2000-333546 20001027  
 JF 2000-333546 20001027  
 JF 2001-249536 20001027  
 KR 2001-711003 20010828  
 KR 2001-711003 20010828  
 HU 2002-3884 20001026  
 US 2002-111479 20020425  
 NO 2002-2007 20020426

## FILING DETAILS:

PATENT NO	KIND	PATENT NO
DE 60003577 E	Based on	EP 1224178 A
KR 433885 B	Previous Publ	KR 2001102429 A
AU 2000079581 A	Based on	WO 2001032637 A
EP 1224178 A1	Based on	WO 2001032637 A
HU 2002003884 A2	Based on	WO 2001032637 A
EP 1224178 B1	Based on	WO 2001032637 A
DE 60003577 E	Based on	WO 2001032637 A
US 6777435 B1	Based on	WO 2001032637 A
KR 433885 B	Based on	WO 2001032637 A

PRIORITY APPLN. INFO: JP 1999-308346 19991029

## INT. PATENT CLASSIF.:

MAIN: C07D; C07D0263-32  
 SECONDARY: A61K0031-42  
 IPC RECLASSIF.: A61K0031-42 [I,A]; A61K0031-421 [I,C]; A61K0045-00 [I,A]; A61K0045-00 [I,C]; A61P0029-00 [I,A]; A61P0029-00 [I,A]; A61P0029-00 [I,C]; A61P0029-00 [I,C]; A61P0003-00 [I,C]; A61P0003-00 [I,C]; A61P0003-06 [I,A]; A61P0003-06 [I,A]; A61P0003-10 [I,A]; A61P0003-10 [I,A]; A61P0043-00 [I,A]; A61P0043-00 [I,C]; A61P0005-00 [I,C]; A61P0005-48 [I,A]; A61P0009-00 [I,C]; A61P0009-10 [I,A]; C07D0263-00 [I,C]; C07D0263-00 [I,C]; C07D0263-32 [I,A]; C07D0263-32 [I,A]

ECLA: C07D0263-32  
 ICO: M07D0263:32  
 USCLASS NCLM: 514/374.000  
 NCLS: 548/236.000

## JAP. PATENT CLASSIF.:

MAIN/SEC.: A61K0031-421; A61K0045-00; A61P0029-00; A61P0003-06; A61P0003-10; A61P0043-00; A61P0005-48; A61P0009-10  
 101; C07D0263-32

FTERM CLASSIF.: 4C056; 4C084; 4C086; 4C201; 4C206; 4C056/AA01;

4C086/AA01; 4C086/AA02; 4C086/AA03; 4C084/AA17;  
 4C056/AB01; 4C056/AC02; 4C056/AD01; 4C056/AE03;  
 4C056/BA03; 4C056/BA08; 4C056/BB01; 4C056/BC01;  
 4C086/BC69; 4C084/DC50; 4C086/MA01; 4C084/MA02;  
 4C086/MA02; 4C086/MA04; 4C084/MA22; 4C086/MA22;  
 4C084/MA23; 4C086/MA23; 4C084/MA31; 4C086/MA31;  
 4C084/MA35; 4C086/MA35; 4C084/MA37; 4C086/MA37;  
 4C084/MA38; 4C086/MA38; 4C084/MA41; 4C086/MA41;  
 4C086/MA43; 4C084/MA52; 4C086/MA52; 4C084/MA55;  
 4C086/MA55; 4C084/MA56; 4C086/MA56; 4C084/MA63;  
 4C084/MA66; 4C086/MA66; 4C086/NA14; 4C086/ZA02;  
 4C086/ZA08; 4C086/ZA15; 4C086/ZA16; 4C086/ZA33;  
 4C086/ZA36; 4C086/ZA40; 4C086/ZA42; 4C086/ZA45;  
 4C086/ZA59; 4C086/ZA66; 4C086/ZA68; 4C086/ZA70;  
 4C086/ZA71; 4C086/ZA75; 4C086/ZA81; 4C086/ZA94;  
 4C086/ZA97; 4C086/ZB11; 4C086/ZB15; 4C086/ZB35;  
 4C086/ZC02; 4C086/ZC23; 4C086/ZC33; 4C084/ZC35.2;  
 4C086/ZC35; 4C086/ZC42; 4C086/ZC54

## BASIC ABSTRACT:

WO 2001032637 A1 UPAB: 20060117

NOVELTY - Crystals of (E)-4-(4-(5-methyl-2-phenyl-4-oxazolylmethoxy)benzyloxyimino)-4-phenylbutyric acid (I) are new, (provided that crystals having a m.pt of 126-127degreesC are excluded).

DETAILED DESCRIPTION - An INDEPENDENT CLAIM is also included for crystals of (I) showing diffraction patterns having characteristic peaks at the spacings (d values) of about 17.5 and about 6.0 Angstrom, by a powder x-ray crystal diffraction.

ACTIVITY - Antidiabetic; Antilipemic; Antiinflammatory; Antiarteriosclerotic; Neuroprotective; Nephrotropic; Ophthalmological; Osteopathic; Antibacterial; Cerebroprotective; Anorectic; Immunomodulator; Hypotensive; Gynecological; Cardiant; Cytostatic; Antidiarrheic; Antirheumatic; Antiarthritic; Antianginal; Antigout; Analgesic; Hepatotropic; Virucide; Antiemetic; Antiulcer; Anti-HIV; Nootropic; Antiparkinsonian; Vasotropic; Antianemic; Depilatory.

The crystals were suspended in a 0.5% methyl cellulose solution (1 mg/ml) and compulsorily administered orally at 1 mg/kg/day to Wistar fatty rats (27-30 weeks old, 5 rats/group), a model of obesity and type 2 diabetes mellitus, for 7 days successively. During that period, diet and water were given freely. Blood was sampled from tail vein and measurement of the components was carried out using serum. The results showed that the crystals showed a hypoglycemic action of 60 % and a hypolipemic action of 85 % compared to controls.

MECHANISM OF ACTION - Retinoid-related receptor agonists and antagonists.

USE - The crystals of (I) can be used in compositions to prevent or treat diabetes mellitus, hyperlipemia, impaired glucose tolerance, inflammatory diseases and arteriosclerosis (claimed). They can also be used as a retinoid-related receptor function regulating agent (claimed). The agent may be a ligand for peroxisome proliferator-activated receptors or for retinoid X receptors (claimed). The crystals of (I) can also be used as an insulin sensitivity enhancing agent or as an insulin resistance improving agent (claimed). The crystals can also be used as agents for preventing or treating diabetic complications (e.g. neuropathy, nephropathy, retinopathy, cataract, macroangiopathy, osteopenia, diabetic hyperosmolar coma, infectious diseases (e.g. respiratory infection, urinary tract infection, gastrointestinal tract infection, dermal soft tissue infection, inferior link infection), diabetic gangrene, xerostomia, lowered sense of hearing, cerebrovascular disease, peripheral circulatory disturbance), obesity, osteoporosis, cachexia (e.g. carcinomatous cachexia, tuberculous cachexia, diabetic cachexia, hemopathic cachexia, endocrinopathic cachexia, infectious cachexia, cachexia induced by

AIDS), fatty liver, hypertension, polycystic ovary syndrome, renal diseases (e.g. diabetic nephropathy, glomerular nephritis, glomerulosclerosis, nephritic syndrome, hypertensive nephrosclerosis, terminal renal disorder), muscular dystrophy, myocardial infarction, angina pectoris, cerebrovascular disease (e.g. cerebral infarction and apoplexy), insulin resistant syndrome, syndrome X, hyperinsulinemia, hyperinsulinemia-induced sensory disorder, tumor (e.g. leukemia, breast, prostate and skin cancer), irritable intestine syndrome, acute or chronic diarrhea, inflammatory disease (e.g. chronic rheumatoid arthritis, spondylitis deformans, osteoarthritis, lumbago, gout, postoperative or traumatic inflammation, remission of swelling, neuralgia, pharyngolaryngitis, cystitis, hepatitis (including steatohepatitis such as nonalcoholic steatohepatitis), pneumonia, pancreatitis, inflammatory colitis, ulcerative colitis), arteriosclerosis (e.g. atherosclerosis), visceral obesity syndrome. They can also be used for ameliorating bellyache, nausea, vomiting, or dysphoria in epigastrium, accompanied by gastrointestinal ulcer, acute or chronic gastritis, biliary dyskinesia or cholecystitis. They can control (enhance or inhibit) appetite and food intake, and be used as an agent for treating leanness and cibophobia or obesity. They can also be used for treating diseases such as viral diseases (AIDS, fulminant hepatitis), neurodegenerative diseases (Alzheimer's and Parkinson's disease, amyotrophic lateral sclerosis, retinitis pigmentosa, cerebellar degeneration), myelodysplasia (e.g. aplastic anemia), ischemic diseases (e.g. myocardial infarction, cerebral apoplexy), hepatic diseases (e.g. alcoholic hepatitis, hepatitis B, hepatitis C, joint-diseases (e.g. osteoarthritis), and atherosclerosis. They can be used for reducing visceral fats, inhibiting accumulation of visceral fats, ameliorating glycometabolism, ameliorating lipid metabolism, ameliorating insulin resistance, inhibiting production of oxidized low density lipoprotein, ameliorating lipoprotein metabolism, ameliorating coronary artery metabolism, preventing or treating cardiovascular complications, preventing or treating heart failure complications, lowering blood remnant, preventing or treating anovulation, hirsutism or hyperandrogenism.

**ADVANTAGE** - The crystals of compound A have excellent physical/chemical properties (e.g. m. pt, solubility, stability) and biological properties (e.g. pharmacokinetics (absorption, distribution, metabolism, excretion, and pharmacodynamics). **TECHNOLOGY FOCUS:**

**PHARMACEUTICALS** - Preferred Drug: The crystals have a m.pt of 136-139degreesC.

**Preparation:** The crystals may be prepared by dissolving (I) in an appropriate solvent (e.g. methanol, or ethanol) at 20-120degreesC, and cooling the resulting solution to 0-50degreesC, preferably 0-20degreesC. (I) may be prepared as in WO99/58510.

#### EXTENSION ABSTRACT:

**ADMINISTRATION** - The crystals of (I) can be used in doses of e.g. 0.05-100, preferably 0.2-4 mg/kg body weight. They can be administered by e.g. oral, parenteral or topical routes.

**EXAMPLE** - Sodium hydride (60 % oily, 3.0 g) was added under nitrogen atmosphere to a solution of methyl (E)-4-hydroxyimino-4-phenylbutyrate (15.5 g) and 4-(4-chloromethylphenoxymethyl)-5-methyl-2-phenyloxazole (23.5 g) in N,N-dimethylformamide (100 ml) at 0degreesC and the mixture was stirred for 2 hours. This was neutralized with 1 N hydrochloric acid, aqueous solution of sodium bicarbonate was added and the mixture extracted with ethyl acetate. The ethyl acetate layer was washed with a saturated aqueous solution of sodium chloride, dried over magnesium sulfate, and concentrated. The residue was subjected to silica gel column chromatography and an oily product was obtained from an eluate with ethyl acetate-hexane (1:3). This was dissolved in tetrahydrofuran (100 ml)-methanol (50 ml), a 2 N aqueous solution of sodium hydroxide (50

ml) was added and the mixture stirred at room temperature for 2 hours. The reaction mixture was neutralized with 1 N hydrochloric acid and extracted with ethyl acetate. The ethyl acetate layer was washed with a saturated aqueous solution of sodium chloride, dried over magnesium sulfate, and concentrated. The resulting crystals were recrystallized from ethyl acetate-hexane to give colorless crystals of (E)-4-(4-(5-methyl-2-phenyl-4-oxazolylmethoxy)benzyloxyimino)-4-phenylbutyric acid (24.5 g, yield 86 %), m. pt 126-127degreesC. A part (500 mg) of the crystals was dissolved in ethanol (5 ml) with heating and cooled at 0degreesC. The precipitating crystals were collected to give crystals (466 mg) showing a powder X-ray crystal diffraction pattern, m. pt 137-138degreesC.

FILE SEGMENT: CPI  
 MANUAL CODE: CPI: B07-E02; B14-A02A5; B14-A02B3; B14-C02; B14-C03;  
 B14-C09; B14-E02; B14-E06; B14-E10; B14-E11; B14-E12;  
 B14-F01E; B14-F02; B14-F02B; B14-F06; B14-F07;  
 B14-G01B; B14-H01; B14-J01A; B14-J01A3; B14-J01A4;  
 B14-K01; B14-N01; B14-N03; B14-N12; B14-S04

L41 ANSWER 3 OF 4 WPIX COPYRIGHT 2009 THOMSON REUTERS on STN  
 ACCESSION NUMBER: 2000-505905 [45] WPIX  
 DOC. NO. CPI: C2000-151857 [45]  
 TITLE: Preparation of membrane vesicles, useful e.g. for  
 diagnosis or treatment of cancer, includes  
 purification by anion-exchange chromatography  
 B04; D16; P34  
 DERWENT CLASS:  
 INVENTOR: AMIGORENA S; CROUZET J; DHELLIN O; RAMEAU P  
 PATENT ASSIGNEE: (ANOS-N) ANOSYS INC; (APCE-N) AP CELLS INC; (INRM-C)  
 INSERM INST NAT SANTE & RECH MEDICALE; (CURI-N) INST  
 CURIE; (INRM-C) INST NAT SANTE & RECH MEDICALE  
 COUNTRY COUNT: 89

## PATENT INFORMATION:

PATENT NO	KIND	DATE	WEEK	LA	PG	MAIN IPC
WO 2000044389	A2	20000803	(200045)*	FR	53	[11]
FR 2788780	A1	20000728	(200045)	FR		
AU 2000030561	A	20000818	(200057)	EN		
EP 1143982	A2	20011017	(200169)	FR		
CN 1355704	A	20020626	(200263)	ZH		
JP 2002535665	W	20021022	(200301)	JA	53	
AU 768322	B	20031211	(200404)	EN		
US 6899863	B1	20050531	(200536)	EN		

## APPLICATION DETAILS:

PATENT NO	KIND	APPLICATION	DATE
WO 2000044389	A2	WO 2000-FR105	20000119
FR 2788780	A1	FR 1999-886	19990127
AU 2000030561	A	AU 2000-30561	20000119
AU 768322	B	AU 2000-30561	20000119
CN 1355704	A	CN 2000-802903	20000119



EP 1143982 A2	EP 2000-900609 20000119
JP 2002535665 W	JP 2000-595691 20000119
EP 1143982 A2	WO 2000-FR105 20000119
JP 2002535665 W	WO 2000-FR105 20000119
US 6899863 B1	WO 2000-FR105 20000119
US 6899863 B1	US 2001-890319 20010726

## FILING DETAILS:

PATENT NO	KIND	PATENT NO
AU 768322 B	Previous Publ	AU 2000030561 A
AU 2000030561 A	Based on	WO 2000044389 A
EP 1143982 A2	Based on	WO 2000044389 A
JP 2002535665 W	Based on	WO 2000044389 A
AU 768322 B	Based on	WO 2000044389 A
US 6899863 B1	Based on	WO 2000044389 A

PRIORITY APPLN. INFO: FR 1999-886

19990127

## INT. PATENT CLASSIF.:

MAIN: G01N0030-88

IPC RECLASSIF.: A61K0035-12 [I,A]; A61K0035-12 [I,C]; A61K0039-00 [I,A]; A61K0039-00 [I,C]; A61K0009-127 [I,A]; A61K0009-127 [I,C]; A61P0035-00 [I,A]; A61P0035-00 [I,C]; B01D0015-08 [I,A]; B01D0015-08 [I,C]; B01J0041-00 [I,C]; B01J0041-04 [I,A]; B01J0041-20 [I,A]; B01J0041-20 [I,C]; C12N0005-08 [I,A]; C12N0005-08 [I,C]; C12P0001-00 [I,A]; C12P0001-00 [I,C]; G01N0030-00 [I,C]; G01N0030-88 [I,A]

ECLA: A61K0035-12; A61K0039-00D6; B01D0015-08; B01J0041-20

ICO: K61K0039:515B

USCLASS NCLM: 424/001.210

NCLS: 424/277.100; 424/520.000; 424/529.000; 424/534.000; 435/317.100

## JAP. PATENT CLASSIF.:

MAIN/SEC.: A61K0035-12; A61K0039-00 H; A61K0009-127;  
A61P0035-00; B01J0041-04 J; C12P0001-00 Z;  
G01N0030-88 E

FTERM CLASSIF.: 2G063; 4B064; 4C076; 4C085; 4C087; 4C201; 4C206;  
4G067; 4C087/AA01; 4C085/AA03; 4C087/AA04;  
4C087/AA05; 4C076/AA19; 4B064/AH19; 4C085/BA01;  
4C087/BB63; 4C087/BB64; 4B064/CA10; 4C085/CC03;  
4C085/CC04; 4C076/CC50; 4B064/CE06; 4B064/CE07;  
4B064/CE11; 4B064/CE12; 4B064/DA01; 4B064/DA13;  
4C085/DD21; 4C085/DD32; 4C076/FF11; 4C076/GG42;  
4C076/GG45; 4C087/MA24; 4C087/NA03; 4C087/NA10

## BASIC ABSTRACT:

WO 2000044389 A2 UPAB: 20060116

NOVELTY - Preparation of membrane vesicles (MV) from a biological sample by at least one step of anion-exchange chromatography (AEC).

DETAILED DESCRIPTION - INDEPENDENT CLAIMS are also included for the following:

- (1) preparation of MV by enriching MV from a sample then treatment by AEC and/or gel permeation chromatography (GPC);
- (2) production of MV by culturing appropriate cells under conditions that cause release of MV, treatment of the culture supernatant to produce a sample enriched in MV (by ultrafiltration or affinity chromatography, AC) then treatment by AEC and/or GPC;
- (3) production of MV by culturing dendritic cells, then purification of MV produced by at least one stage of AEC;

- (4) use of AEC or AC for preparation or purification of MV; and  
(5) compositions containing MV produced by the new methods.

ACTIVITY - Cytostatic; immunomodulator.

No supporting biological data is given.

MECHANISM OF ACTION - None given.

USE - MV are used in diagnosis, vaccination and therapy, also for delivery of selected compounds, e.g. for study or treatment of cancer; for regulating the immune system; for preparation of antibodies; for labeling and for constructing banks of materials. The method can also be used to detect contamination in batches of MV.

ADVANTAGE - The method allows large scale production/purification of MV for pharmaceutical use, and can be applied to autologous MV or those produced by established cell lines. AEC resolves MV as a single, homogeneous peak, despite the complex nature of MV, so provides efficient removal of contaminating proteins and nucleic acids.

#### TECHNOLOGY FOCUS:

BIOLOGY - Preferred Method: This includes at least one step of AEC on a strong anion resin, optionally also at least one step of GPC. The sample is a biological fluid, culture supernatant, cell lysate or pre-purified solution. The method particularly includes preliminary culture under conditions that cause release of MV and enrichment involving clarification (by low-speed centrifugation and/or filtration) and optionally concentration, preferably by AC on a dye-containing material. The optional concentration step is alternatively by ultrafiltration, particularly tangential. The collected material may be filtered (particularly through a 0.22 micron membrane). AEC is particularly performed under pressure and columns are eluted with a gradient of 0-2 M sodium chloride, with MV recovered at 350-700 mM.

Preferred Materials: AEC is performed on conventional supports, preferably formed as beads of high porosity, especially of pore size 0.1-1 micron. Preferred materials include poly(styrene-divinylbenzene), polyacrylamide, agarose, dextran and/or silica, functionalized by quaternary ammonium groups. Most preferred is 'Source Q15'. The optional GPC step is performed after AEC, especially using 'Superdex 200HR' as support. AC is typically on Blue Sepharose. Preferred vesicles: MV have diameter 60-90 nm and are produced by antigen-presenting cells, particularly dendritic cells, B lymphocytes, macrophages and mastocytes, best human dendritic cells. Alternatively MV are produced by tumor cells. The preferred source of MV is dendritic cells, optionally immature cells from bone marrow or peripheral blood. These cells may be sensitized to antigens before preparation of MV (e.g. by incubation with peptides, nucleic acid or antigens), cultured under conditions that favor production of MV (e.g. addition of gamma-interferon or an interleukin), immortalized and genetically modified to express, at the surface of MV, selected proteins or developed from their monocyte precursors by treatment with granulocyte-macrophage colony-stimulating factor and interleukin-4 or -13.

#### EXTENSION ABSTRACT:

EXAMPLE - Cultures of the TS/A tumor cell line were centrifuged to isolate exosomes, then these suspended in 0.1 ml saline to protein concentration 0.5-1 mg/ml. 40 microliters of the product, diluted in 0.5 ml Tris-hydrochloride, were injected on to a 'Source Q15' column, equilibrated with similar buffer of pH 8. The column was eluted with 30 column volumes of a 0-0.5 M sodium chloride gradient, then with 2 M sodium chloride. Monitoring of the eluate at 260-280 nm indicated 3 sharp peaks at 0.105, 0.4 and 2 M. The 0.4 M peak had protein profile identical to that of exosomes

produced by the classical centrifugation method.

FILE SEGMENT: CPI; GMPI  
 MANUAL CODE: CPI: B04-F01; B11-C08D2; B12-K04; B14-G03; B14-H01B;  
 B14-S11; D05-H07; D05-H09

L41 ANSWER 4 OF 4 WPIX COPYRIGHT 2009 THOMSON REUTERS on STN  
 ACCESSION NUMBER: 1989-294134 [41] WPIX  
 DOC. NO. CPI: C1989-130216 [21]  
 TITLE: Purificn. of 5'-flavin mono:nucleotide salts - by  
 chromatography on adsorption resin and/or  
 alkyl-derivatised silica gel  
 DERWENT CLASS: B02; B03; C02; D13; E11  
 INVENTOR: DOBLER W; EGGERSDORF M; EGGERSDORFER M; PAUST J  
 PATENT ASSIGNEE: (BADI-C) BASF AG  
 COUNTRY COUNT: 8

## PATENT INFORMATION:

PATENT NO	KIND	DATE	WEEK	LA	PG	MAIN IPC
EP 336214	A	19891011	(198941)*	DE	11[0]	
<--						
DE 3810957	A	19891019	(198943)	DE		
<--						
JP 01287084	A	19891117	(199001)	JA		
<--						
US 4987229	A	19910122	(199106)	EN		
<--						
EP 336214	B	19920401	(199214)	EN	12	
<--						
DE 58901065	G	19920507	(199220)	DE		
<--						
JP 2856760	B2	19990210	(199911)	JA	7	
<--						

## APPLICATION DETAILS:

PATENT NO	KIND	APPLICATION	DATE
EP 336214 A		EP 1989-105097	19890322
DE 3810957 A		DE 1988-3810957	19880331
DE 58901065 G		DE 1988-3810957	19880331
US 4987229 A		US 1989-323795	19890315
EP 336214 B		EP 1989-105097	19890322
JP 01287084 A		JP 1989-78692	19890331
JP 2856760 B2		JP 1989-78692	19890331

## FILING DETAILS:

PATENT NO	KIND	PATENT NO
JP 2856760 B2	Previous Publ	JP 01287084 A

PRIORITY APPLN. INFO: DE 1988-3810957 19880331  
 INT. PATENT CLASSIF.:  
 SECONDARY: C07F0009-54  
 IPC RECLASSIF.: C07D0475-00 [I,C]; C07D0475-14 [I,A]; C07F0009-00  
 [I,C]; C07F0009-6561 [I,A]  
 ECLA: C07F0009-6561F  
 USCLASS NCLM: 544/243.000

NCLS: 544/244.000; 987/050.000  
JAP. PATENT CLASSIF.:  
MAIN/SEC.: C07D0475-14  
FTERM CLASSIF.: 4C049; 4C049/FF01; 4C049/GG05  
BASIC ABSTRACT:

EP 336214 A UPAB: 20050429

Production of 5'-flavin mononucleotide (5'-FMN) salts is effected by:  
(a) phosphorylating riboflavin (RF) to form crude 5'-FMN contaminated with RF and isomeric RF mono- and diphosphates; (b) reacting the crude 5'-FMN with an alkali metal hydroxide or nitrogen base in H<sub>2</sub>O, opt. with heating to 30-100 deg. C, to form a homogeneous clear 5'-FMN salt solution with a concentration of 1-5% and a pH of 4-8; (c) contacting the solution with an adsorption resin comprising spherical particles of a copolymer of styrene or (meth)acrylic acid derivs. with divinylbenzene; and (d) isolating the RF-free 5'-FMN salt from the solution

Purificn. of RF-free 5'-FMN monosodium salt contaminated with isomeric flavin mono- and dinucleotides is effected by chromatographing a 1-15 (especially 4-6) weight% solution of the crude salt in a solvent comprising 20-100 weight% H<sub>2</sub>O and 0-80 weight% of a lower aliphatic alcohol on an alkyl-derivatised RP silica gel with a particle size of 10-300 microns, eluting with a solvent of the same type. The solution has a pH of 4-8 and is applied in an amount of at least 5-50% of the bed volume of the silica gel column.

USE - 5'-FMN is useful as an additive for medicaments, foods and animal feeds. Na 5'-FMN is an intermediate for flavin adenine dinucleotide, which is useful for treating vitamin B2 deficiency.

FILE SEGMENT: CPI  
MANUAL CODE: CPI: B03-C; B12-J01; C03-C; C12-J01; D03-G01;  
D03-H01; E03; E06-D17

=> FIL DISSABS PASCAL

FILE 'DISSABS' ENTERED AT 09:23:03 ON 17 DEC 2009

COPYRIGHT (C) 2009 ProQuest Information and Learning Company; All Rights Reserved.

FILE 'PASCAL' ENTERED AT 09:23:03 ON 17 DEC 2009

Any reproduction or dissemination in part or in full,  
by means of any process and on any support whatsoever  
is prohibited without the prior written agreement of INIST-CNRS.  
COPYRIGHT (C) 2009 INIST-CNRS. All rights reserved.

=> D L152 1-5 IALL

L152 ANSWER 1 OF 5 DISSABS COPYRIGHT (C) 2009 ProQuest Information and Learning Company; All Rights Reserved on STN

ACCESSION NUMBER: 87:7906 DISSABS Order Number: AAR8714466  
TITLE: THE SYNTHESIS OF NON-POROUS SILICA  
SUPPORTS AND THEIR APPLICATION TO THE SEPARATION OF  
PROTEINS BY HIGH PERFORMANCE LIQUID  
CHROMATOGRAPHY  
AUTHOR: COLWELL, LAWRENCE F., JR. [PH.D.]  
CORPORATE SOURCE: RUTGERS THE STATE UNIVERSITY OF NEW JERSEY - NEW  
BRUNSWICK (0190)  
SOURCE: Dissertation Abstracts International, (1987)  
Vol. 48, No. 3B, p. 727. Order No.: AAR8714466. 235  
pages.  
DOCUMENT TYPE: Dissertation  
FILE SEGMENT: DAI

LANGUAGE: English  
ENTRY DATE: Entered STN: 19921118  
Last Updated on STN: 19921118  
ABSTRACT: The synthesis of non-porous

silica supports and their application to the reversed-phase HPLC separation of proteins is described. Non-porous micro-spheres with diameters below 2 microns were synthesized via the basic hydrolysis of n-alkyltetraortho silicates and the subsequent polymerization of the resulting silicic acid. The size of the particles produced was a function of the reaction conditions and the length of the n-alkyl carbon chain. Non-porous particles with diameters above 2 microns were produced by plugging the pores of commercial HPLC supports, using reaction conditions similar to those employed to make the solid spheres. Simple bench-top procedures were developed that permitted the in-house measurement of both particle diameter and surface area. A stirred-slurry reservoir was used to pack very efficient HPLC columns. A series of non-porous silica supports was prepared and compared to conventional porous supports. The fastest reversed-phase separation of proteins was achieved with a 10 micron non-porous

support; the greatest resolution was obtained with sub-micron solid spheres. The results of this study indicate that porous supports are unnecessary for reversed-phased protein separations, and non-porous supports provide a superior alternative to the present technology.

CLASSIFICATION: 0485 CHEMISTRY, GENERAL

L152 ANSWER 2 OF 5 DISSABS COPYRIGHT (C) 2009 ProQuest Information and Learning Company; All Rights Reserved on STN  
ACCESSION NUMBER: 82:28970 DISSABS Order Number: AAR8310847  
TITLE: DEVELOPMENT OF SUPPORT MATERIALS FOR HIGH - PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC, SILICA, POLYETHYLENEIMINE)  
AUTHOR: PEARSON, JAMES DAVID [PH.D.]  
CORPORATE SOURCE: PURDUE UNIVERSITY (0183)  
SOURCE: Dissertation Abstracts International, (1982) Vol. 43, No. 12B, p. 3960. Order No.: AAR8310847. 270

pages.  
DOCUMENT TYPE: Dissertation  
FILE SEGMENT: DAI  
LANGUAGE: English  
ENTRY DATE: Entered STN: 19921118  
Last Updated on STN: 19921118  
ABSTRACT: Large pore ((GREATERTHEQ) 300 (ANGSTROM

)), spherical microparticulate

silicas possessing n-alkyl hydrocarbon ligands were found to be optimal for the purification of large denatured peptides. Various macroporous silicas were tested and it was found that some silicas offered better selectivity than others for reversed-phase peptide and protein separations. Additional variables found to affect protein resolution were thermal pretreatment of silica, silylation reaction conditions, and n-alkyl chain length. A test mixture for monitoring protein resolution potential was established because theoretical plate values were poor indicators of column performance. Peptides and proteins were nicely resolved on columns only 5 cm in length. The interaction between protein and stationary phase was based on an adsorption rather than partition mechanism.

A method for fractionating transfer ribonucleic acids by descending salt gradient elution on reversed-phase columns was developed.

Stationary phase variables such as n-alkyl chain length and silica type were investigated and it was concluded that ethyl or n-butyl groups covalently attached to macroporous silica

worked the best. Mobile phase parameters including salt concentration, organic solvent, pH and temperature were found to affect resolution.

Polyethyleneimine anion-exchangers were developed for both protein and oligonucleotide fractionations.

The process consisted of adsorbing polyethyleneimine onto silica and then covalently immobilizing the polyamine layer with a bifunctional crosslinker. The type of crosslinker employed was found to contribute to stationary phase selectivity for both proteins and mononucleotides. Changes in ionic strength could be used to alter retention times of various proteins on anion-exchangers depending upon the relative lipophilicity of the crosslinking agent.

Development of oligonucleotide anion-exchangers based on polyethyleneimine was also achieved and a general method for converting pre-packed silica columns into anion-exchangers was outlined.

Short, 5 cm anion-exchange columns were

capable of fractionating oligonucleotide homologs of up to at least 35 bases in chain length.

CLASSIFICATION: 0487 CHEMISTRY, BIOCHEMISTRY; 0486 CHEMISTRY, ANALYTICAL

L152 ANSWER 3 OF 5 PASCAL COPYRIGHT 2009 INIST-CNRS. ALL RIGHTS RESERVED. on STN

ACCESSION NUMBER: 2000-0466282 PASCAL Full-text

COPYRIGHT NOTICE: Copyright .COPYRGT. 2000 INIST-CNRS. All rights reserved.

TITLE (IN ENGLISH): Synthesis of spherical porous silicas in the micron and submicron size range : challenges and opportunities for miniaturized high-resolution chromatographic and electrokinetic separations  
Analytical Separations 2000, Vaals, Netherlands, 13-16 October 1999

AUTHOR: UNGER K. K.; KUMAR D.; GRIM M.; BUECHEL G.; LUEDTKE S.; ADAM Th.; SCHUMACHER K.; RENKER S. POOLE C. F. (ed.)

CORPORATE SOURCE: Institut fuer Anorganische und Analytische Chemie, Johannes Gutenberg - Universitaet, Duesbergweg 10-14, 55099 Mainz, Germany, Federal Republic of

SOURCE: Journal of chromatography, {2000}, 892(1-2), 47-55, 39 refs.

Conference: Analytical Separations 2000, Vaals (Netherlands), 13 Oct 1999

ISSN: 0021-9673 CODEN: JOCRAM

DOCUMENT TYPE: Journal; Conference

BIBLIOGRAPHIC LEVEL: Analytic

COUNTRY: Netherlands

LANGUAGE: English

AVAILABILITY: INIST-8577A, 354000091916940040

ABSTRACT: Classical silica technology has reached its limit with respect to an ultimate minimum

particle size of about 2  $\mu\text{m}$  in diameter. Here, a novel process is presented which allows one to synthesize porous silica beads and control their particle diameter in situ, within the range of 0.2-2.0  $\mu\text{m}$ . As a result, no sizing is required and losses of silica are avoided. Furthermore, the process enables one to control in situ the pore structural parameters and the surface chemistry of the silica beads. Even though surface functionalized silicas made according to this process can principally be applied in fast HPLC the column pressure drop will be high even for short columns. In addition, the column efficiency, expressed in terms of the theoretical plate height is about  $H_{\text{eqv}} \sim 2d_{\text{sub}} \cdot \pi$  in the best case and limited by the A and C term of the Van Deemter equation. In other words the gain in total plate number when using 1-2  $\mu\text{m}$  silica beads in short columns is minimal as compared to longer columns packed with 5  $\mu\text{m}$  particles. Capillary electrochromatography (CEC) as a hybrid method enables the application of micron size as well as submicron size particles. This consequently enhances column efficiency by a factor of 5-10 when compared to HPLC. The use of short CEC columns packed with submicron size silicas provides the basis for fast and efficient miniaturized systems. The most significant feature of CEC as compared to HPLC is that the former allows one to resolve polar and ionic analytes in a single run. An alternative method for miniaturization is



capillary electrophoresis (CE) which generates extremely high efficiencies combined with fast analysis. Its application, however, is limited to ionic substances.

CLASSIFICATION CODE: 001C04B02; Chemistry; Analytical chemistry; Chromatography  
CONTROLLED TERM: Review; Stationary phase; Silica gel; Micron size particle; Submicron particle; Porous material; Spherical particle; HPLC chromatography; Capillary electrophoresis; Electrochromatography; Miniaturization; Chromatographic retention; Digitoxin; Cardenolide; Steroid; Aglycone; Glycoside; Cardiotoxic agent

L152 ANSWER 4 OF 5 PASCAL COPYRIGHT 2009 INIST-CNRS. ALL RIGHTS RESERVED. on STN

ACCESSION NUMBER: 1999-0318018 PASCAL Full-text  
COPYRIGHT NOTICE: Copyright .COPYRG. 1999 INIST-CNRS. All rights reserved.

TITLE (IN ENGLISH): Packing and stationary phase design for capillary electroendosmotic chromatography (CEC)  
Symposium on High Performance Liquid Phase Micro Separations, Lund (Sweden), June 7-10, 1998

AUTHOR: ADAM T.; LUEDTKE S.; UNGER K. K.  
CORPORATE SOURCE: Institut fuer Anorganische und Analytische Chemie, Johannes Gutenberg - Universitaet, 55099 Mainz, Germany, Federal Republic of Swedish Academy of Pharmaceutical Sciences, Sweden (patr.); Lund University, Sweden (patr.)

SOURCE: Chromatographia, (1999), 49(SUP1), 49-55, 20 refs.  
Conference: Symposium on High Performance Liquid Phase Micro Separations, Lund (Sweden), 7 Jun 1998  
ISSN: 0009-5893 CODEN: CHRG7

DOCUMENT TYPE: Journal; Conference  
BIBLIOGRAPHIC LEVEL: Analytic  
COUNTRY: Germany, Federal Republic of  
LANGUAGE: English  
AVAILABILITY: INIST-8489, 354000083681290070  
ABSTRACT: Reversed-phase stationary phases based on

porous silica beads in

the range 0.2-3  $\mu\text{m}$  have been prepared to study the dependency of electroosmotic flow (EOF) on particle diameter. Additionally, the influence of the mobile phase composition, i.e. organic modifier content, pH of buffer solution and concentration of buffer salt, on the magnitude of the electroosmotic flow velocity has been investigated. To perform separations in an aqueous acidic mobile-phase, mixed mode stationary phases containing both alkyl chains and strong

cation-exchange groups have been applied.

CLASSIFICATION CODE: 001C04B02; Chemistry; Analytical chemistry; Chromatography

CONTROLLED TERM: Experimental study; Bonded stationary phase; Packed column; Submicron particle; Silica gel; Porous material; Particle size; Phase composition; Mobile phase; pH; Electrochromatography; Reversed phase chromatography; Cation exchanger; Peak resolution; Efficiency; Organic compounds

L152 ANSWER 5 OF 5 PASCAL COPYRIGHT 2009 INIST-CNRS. ALL RIGHTS RESERVED. on STN

ACCESSION NUMBER: 1999-0140545 PASCAL Full-text

COPYRIGHT NOTICE: Copyright .COPYRGT. 1999 INIST-CNRS. All rights reserved.

TITLE (IN ENGLISH): Ultrahigh-pressure reversed-phase capillary liquid chromatography : Isocratic and gradient elution using columns packed with 1.0- $\mu$ m particles

AUTHOR: MACNAIR J. E.; PATEL K. D.; JORGENSON J. W.

CORPORATE SOURCE: Department of Chemistry, University of North Carolina at Chapel Hill, Venable Hall, CB 3290, Chapel Hill, North Carolina 27599-3290, United States

SOURCE: Analytical chemistry : (Washington, DC), {1999}, 71(3), 700-708, 16 refs. ISSN: 0003-2700 CODEN: ANCHAM

DOCUMENT TYPE: Journal

BIBLIOGRAPHIC LEVEL: Analytic

COUNTRY: United States

LANGUAGE: English

AVAILABILITY: INIST-120B, 354000073769170240

ABSTRACT: Fused-silica capillaries with inner

diameters of 33  $\mu$ m and lengths of 25-50 cm are

slurry-packed with 1.0- $\mu$ m nonporous

octadecylsilane-modified (C.sub.1.sub.8)

**silica spheres. These**

columns are used to perform

ultrahigh-pressure reversed-phase liquid

chromatographic analyses in both isocratic

and gradient elution modes. Mobile-phase pressures

as high as 5000 bar (72 000 psi) are applied to

column inlets to generate more than 200

000 theoretical plates in 6 min ( $k'$  1) for small,

organic analytes. Average capacity factors of

analytes are found to increase linearly with

applied pressure. An electrically driven

constant-flow syringe pump capable of generating mobile-phase pressures as high as 9000 bar (130 000 psi) is described. This pump is used in conjunction with an exponential dilution method for the gradient separation of peptides from a tryptic digest on a 27-cm-long capillary packed with 1.0- $\mu$ m particles. A peak capacity of 300 is demonstrated for a 30-min analysis.

CLASSIFICATION CODE: 001C04B02; Chemistry; Analytical chemistry; Chromatography

CONTROLLED TERM: Optimization; Operating conditions; Isocratic condition; Peak resolution; Analysis method; Separation method; Reversed phase chromatography; HPLC chromatography; Fluorescence detector; Column packing; Solid particle; Micron size particle; High pressure; Performance evaluation